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ANNUAL SUMMARY REPORT

on

DEVELOPMENT OF A RAPID STRESS
CORROSION TEST FOR ALUMINUM ALLOYS

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

March 15, 1967

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Gentlemen:

Contract No. NAS8-20285
Control No. DCN 1-6-54-01045 (1F)

Enclosed are seventeen copies of the Annual Summary Report on "Development of a Rapid Stress Corrosion Test for Aluminum Alloys", covering work performed during the period 1 March 1966 to 1 March 1967. Distribution of these copies has been made as noted below.

Very truly yours,



T. R. Pritchett
Laboratory Manager

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FOREWORD

This report was prepared by Kaiser Aluminum & Chemical Corporation, Department of Metallurgical Research, Spokane, Washington, under Contract No. NAS8-20285, "Development of a Rapid Stress Corrosion Test for Aluminum Alloys", for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. The work was administered under the technical direction of the Propulsion and Vehicle Engineering Laboratory, Materials Division of the George C. Marshall Space Flight Center with T. S. Humphries acting as project manager.

This report covers work done from 1 March 1966 through 1 March 1967.

A total of 4112 man-hours was expended in accomplishing the work performed during this report period.

Kaiser research personnel who made major contributions to the program were: W. J. Helfrich, project leader; T. J. Summerson, research chemist; D. A. Jones, research chemist; J. C. Regis, project technician; and Frances Johnston, metallographer. This report is identified internally as MS PR 67-51. The data reported herein are recorded in DMR Laboratory Record Books Nos. 1470, 1716, 1725, 1731, 1736, 1765, 1774, 1785, 1797 and 1902.

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ABSTRACT

Various media were investigated for use in accelerated stress corrosion tests of aluminum alloys. In general, aqueous solutions of $\text{NaCl-K}_2\text{Cr}_2\text{O}_7$ were very effective in causing rapid short-transverse failures in the absence of excessive general surface corrosion. However, the stress corrosion performance of any one alloy and temper was dependent upon the solution pH and temperature. Acid solutions ($\text{pH} \leq 1.5$) promoted rapid stress corrosion cracking at room temperature, but they were far too aggressive for a practical test. Less aggressive media, ca. $\text{pH} = 2$ to 4 , were effective in accelerating failures of 7039-T64, -T61 and 7075-T6, but not effective in tests of 2024-T4, -T351 and 2219-T42. In fact, the rate of cracking in these 2000 series alloys at a $\text{pH} = 3$ and temperature of 30°C was no more rapid than that observed in 3.5% NaCl alternate immersion tests. Increasing the solution temperature at $\text{pH} = 3.7$ accelerated the stress corrosion cracking of 7039-T64 and 2024-T4 and -T351, but cracking was virtually eliminated in 7075-T6 at 60°C or 100°C .

Neutral salt-(di)chromate solutions promoted rapid stress corrosion failures in susceptible tempers of 2024 and 2219. Unfortunately, these media were not very effective on 7075-T6 and ineffective in tests of 7039-T64 and -T61. Stress corrosion of 7039 increased with increasing temperature, however, there was no apparent acceleration in the rate of cracking in 7075-T6.

Neutral salt-(di)chromate solutions are relatively innocuous towards unstressed aluminum alloys. As such, they would be preferred to the more corrosive 3.5% NaCl solution employed in alternate immersion tests. However, to consistently achieve more rapid rates of cracking than that obtained in alternate immersion tests, it was necessary to anodically polarize specimens exposed to a neutral 3% NaCl-2% $\text{K}_2\text{Cr}_2\text{O}_7$ solution at a current density of 50 microamperes/cm². This test shows promise as a method of separating susceptible and resistant tempers of 2024, 2219 and 7075 and duplicating the threshold stresses for stress corrosion of these alloys observed in 3.5% NaCl alternate immersion tests.

However, while intensiostatic stress corrosion tests are easily implemented, they are not amenable to large volume testing. As a result, consideration has been given to modification of the neutral salt-(di)chromate solution with additions of strong oxidizing agents and use of buffered sodium chloride solutions in accelerated stress corrosion tests. These studies will form the basis for future work under an extension of this contract.

OBJECTIVES

The objective of this research is the development of a rapid stress corrosion susceptibility test for aluminum alloys which can be completed in one week or less. Implementation of the test should not require elaborate accessory equipment other than that normally available in metallurgical and chemical research facilities. The applicability of the test will be established (1) through correlation with conventional laboratory and outdoor exposure tests and published data, and (2) by comparison with the threshold stresses obtained in conventional stress corrosion tests and service exposures.

It is also the objective of this research to gain additional understanding of the processes which contribute to stress corrosion cracking in aluminum alloys.

TABLE OF CONTENTS

	Page
INTRODUCTION -----	1
EXPERIMENTAL -----	3
Materials -----	3
Specimens -----	4
Figure 1 C-Ring Stress Corrosion Test Specimens -----	5
Figure 2 Stress Corrosion Test Cell -----	6
Figure 3 Schematic Diagram of Apparatus for Stress Corrosion Testing -----	6
Procedures -----	7
Exploratory Stress Corrosion Tests -----	7
Intensiostatic Tests -----	9
Figure 4 Cell for Intensiostatic Stress Corrosion Tests -----	8
Figure 5 Schematic of Electrical Circuit for Intensiostatic Stress Corrosion Testing-----	8
3.5% NaCl Alternate Immersion Tests -----	10
Atmospheric Exposure Tests -----	10
RESULTS -----	11
1 Exploratory Stress Corrosion Tests of Free-Corrosion Specimens -----	11
pH Dependence -----	11
2024 Alloy -----	11
Figure 6 Effect of pH on the Stress Corrosion Resistance of 2024-T4 in Salt-(Di) Chromate Solutions at 30C -----	13
2219 Alloy -----	14
Figure 7 Effect of pH on Stress Corrosion of 2219-T42 in Salt-(Di)Chromate Solutions at 30C -----	15
7075 Alloy -----	16
Figure 8 Effect of pH on Stress Corrosion of 7075-T651 in Salt-(Di)Chromate Solutions at 30C -----	17

TABLE OF CONTENTS (CONT'D)

	Page
7039 Alloy -----	16
Figure 9 Effect of pH on Stress Corrosion of 7039-T64 in Salt-(Di)Chromate Solutions at 30C -----	17
Temperature Dependence -----	19
2024 Alloy -----	19
Figure 10 Effect of Temperature on Stress Corrosion of 2024-T4 and 2219-T42 in Neutral Salt-(Di)Chromate Solutions -----	20
7075 Alloy -----	21
Figure 11 Effect of Temperature on Stress Corrosion of 7075-T651 in Salt-(Di) Chromate Solutions at pH = 1 and 3.7- -----	23
7039 Alloy -----	22
Figure 12 Effect of Temperature on Stress Corrosion of 7039-T64 (742101) in Salt-(Di)Chromate Solutions at pH = 1, 3.7 and 7 -----	23
II Stress Corrosion Tests of Galvanic Couples -----	24
III Intensiostatic Stress Corrosion Tests -----	26
IV 3.5% NaCl Alternate Immersion Tests -----	27
V Marine and Industrial Atmosphere Tests -----	28
Figure 13 Effect of Environment on the Short- Transverse Stress Corrosion Resistance of Alloy 7039 -----	30
DISCUSSION -----	31
STATUS -----	35
REFERENCES -----	36
TABLE I Chemical Compositions, Tensile Properties, and Conductivities of Experimental Materials -----	38

TABLE OF CONTENTS (CONT'D)

		Page
TABLE II	Effect of pH and Chloride Ion Concentration on Short-Transverse Stress Corrosion Cracking of 2024-T351, -T4, and -T6 Continuously Immersed in Salt-(Di)Chromate Solutions at 30C -----	40
TABLE III	Effect of pH on Short-Transverse Stress Corrosion Cracking of 2219-T42 Continuously Immersed in Salt-(Di)Chromate Solutions at 30C -----	41
TABLE IV	Effect of pH on Short-Transverse Stress Corrosion Cracking of 7075-T651 Continuously Immersed in Salt-(Di)Chromate Solutions at 30C -----	42
TABLE V	Effect of pH on Short-Transverse Stress Corrosion Cracking of 7039-T64 Continuously Immersed in Salt-(Di)Chromate Solutions at 30C -----	43
TABLE VI	Influence of Temperature on Short-Transverse Stress Corrosion Cracking of 7039-T64, -T61 and -T6E132 Subject to Continuous Immersion in 1 Molar NaCl Solution -----	44
TABLE VII	Effect of Temperature on Short-Transverse Stress Corrosion of 2024-T4 and 2219-T42 Subject to Continuous Immersion in Salt-(Di)Chromate Solutions at pH = 1, 3.7 and 7 -----	45
TABLE VIII	Influence of Temperature on Short-Transverse Corrosion of 7075-T651 Subject to Continuous Immersion in Salt-(Di)Chromate Solutions at pH = 1, 3.7 and 7 ---	46
TABLE IX	Effect of Temperature on Short-Transverse Stress Corrosion of 7039-T64 Continuously Immersed in Salt-(Di)Chromate Solutions at pH = 1, 3.7 and 7 --	47
TABLE X	Short-Transverse Stress Corrosion of Copper-Aluminum Alloy Galvanic Couples in a Neutral 5% NaCl - 2% $K_2Cr_2O_7$ Solution at 30C -----	48
TABLE XI	Short-Transverse Stress Corrosion of 2024, 2219 and 7075 Tested Intensiostatically in Neutral 3% NaCl- 2% $K_2Cr_2O_7$ Solution at 30C -----	49
TABLE XII	Short-Transverse Stress Corrosion Cracking of 2024, 2219, 7075 and 7039 in 3.5% NaCl Solution by Alternate Immersion at 75F \pm 2F -----	50

TABLE OF CONTENTS (CONT'D)

	Page
TABLE XIII Short-Transverse Stress Corrosion of 2024, 2219, 7075 and 7039 Alloys in the Marine Atmosphere of Daytona Beach, Florida -----	53
TABLE XIV Short-Transverse Stress Corrosion of 7075 and 7039 in the Industrial Atmosphere of Pittsburgh, Pa.-----	55

DEVELOPMENT OF A RAPID STRESS CORROSION TEST
FOR ALUMINUM ALLOYS

By

W. J. Helfrich

INTRODUCTION

Virtually all of the stress corrosion tests for aluminum alloys involve exposure of appropriate test specimens to the conjoint action of a sustained tensile stress and chloride-containing environment (Ref 1). One popular test consists of cyclic exposure to a 3.5% sodium chloride solution for 10 minutes, followed by 50 minutes of air drying at room temperature. This test has proved very effective in promoting stress corrosion of a variety of highly susceptible aluminum alloys and, in most instances, the results have correlated well with industrial and marine exposures.

Recently, however, new copper-free Al-Zn-Mg alloys, such as 7039, have been made available, providing high strength and excellent general corrosion resistance in welded structures. Correlation of 3.5% NaCl alternate immersion tests with the anticipated stress corrosion performance of these alloys in natural environments has become extremely complex. Lifka and Sprowls (Ref 2) have shown disparate stress corrosion behavior in laboratory, marine, and industrial tests of alloys 7075, 7079 and 7039. Notwithstanding that some of this confusion stems from lack of control over the testing conditions (Ref 3), these results point to a definite need today for more reliable test methods for evaluation of stress corrosion performance.

Greater emphasis is also being placed on more rapid assessment of material performance. In the past few years, special tempers have been introduced in certain high-strength aluminum alloys for the purpose of providing maximum resistance to stress corrosion cracking. Examples are 7075-T73 and 2219-T81 or -T87. Assurance that these items possess good stress corrosion resistance has frequently been defined by alternate immersion tests of thirty days or longer duration. The time required to conduct these tests can result in delay in shipment and use of these materials. As a result, more rapid test

methods, suitable for both plant and user application, are desired to insure that alloys produced in these special tempers possess and maintain high resistance to stress corrosion cracking.

Interest in the development of rapid stress corrosion tests for aluminum alloys is not new. More than 20 years ago, alternate immersion, spraying, and continuous immersion in sodium chloride, sodium chloride-hydrogen peroxide, sodium chloride-sodium chromate, acidified salt-dichromate, dilute sulfuric acid, and boiling sodium chloride solutions were advocated for use in accelerated stress corrosion tests of aluminum alloys (Refs 4 & 5). Except for alternate immersion in 3.5% NaCl solution (Ref 5), subsequently modified to a 10-minute wet / 50-minute drying cycle, these tests have not gained wide acceptance -- not that they differ so radically from service environments (admittedly, some do), but rather that (1) the results obtained have not correlated with outdoor exposures, (2) the tests were specific for one alloy system, or (3) they were either too aggressive or not aggressive enough.

Curiously, however, these tests and others unsuited for assessing stress corrosion performance in service environments have been used in studies of the mechanism of stress corrosion. As a result, it is not surprising to find that standardized tests for approaching this problem do not exist. Testing is a matter of personal experience and philosophy, and, in most instances, more questions than answers have been provided through work of this nature. On the other hand, studies of the basic or rate-controlling processes for stress corrosion are essential to the development of practical test methods. Consequently, our approach involved intensive studies of the environmental factors affecting stress corrosion as a means of eventually specifying the conditions under which tests for a wide range of aluminum alloys could be conducted.

EXPERIMENTAL

Materials

The following alloys and tempers were evaluated:

2024-T351, -T851, -T4, -T6

2219-T42, -T62, -T351, -T851, -T37, -T87

7075-T651, -T7351

7039-T64, -T61, -T6E132

While the C-ring was the only type specimen used in this study, all test items, excepting one lot of 7039-T64, were procured in 2-inch to 3-inch plate thicknesses in anticipation of future evaluation of short-transverse stress corrosion performance using 2-inch by 1/8-inch diameter tensile specimens. Sufficient surface area of plate was purchased to provide approximately 1000 C-rings or other short-transverse test specimens of each alloy and temper.

Excepting the 7039-T6E132*, we obtained sufficient plate from production lots of each basic temper to prepare the related artificially or overaged tempers. This procedure minimized the gross changes in chemistry and fabrication which otherwise may have occurred if various tempers of an alloy were taken from different production lots. The 2024 was obtained in the -T4 and -T351 tempers. Portions of these samples were then artificially aged in the laboratory to the -T6 and -T851 tempers. The mill-fabricated 7075-T651 was similarly sampled and aged to the -T7351 temper. The 2219 was purchased as-fabricated* for further processing at our Trentwood Works. This processing included solution heat treatment and subsequent cold working of the -T42 product approximately 3% and 9% (by stretching) to obtain the -T351 and -T37 tempers, respectively. Artificial aging of these items to the -T62, -T851 and -T87 tempers was performed at our metallurgical laboratories. The 7039-T61 was obtained by re-solution heat treating and artificial aging a portion of the plant-fabricated 7039-T64 plate.

* Aluminum Company of America.

Table I gives the chemical composition, tensile properties, and electrical conductivities (where appropriate) for these 15 test items. We suggest that the data are typical of the respective products and indicative of proper heat treatment in all cases. It must be remembered, however, that the results of our stress corrosion studies must be interpreted in the light of tests performed on one or two lots of each item.

Specimens

The short-transverse C-rings were 0.75 inch wide, with a 0.948-inch outside diameter and a 0.060-inch wall thickness. All specimens were etched for 30 seconds in 5% NaOH solution at 170F and rinsed in hot water. This was followed by desmutting in cold concentrated HNO₃ for 15 to 30 seconds with a final hot water rinse. This procedure gave a quick check of proper short-transverse orientation and a clean water-break-free surface. The etch also removed surface cold work introduced during the machining of the specimens.

After stressing, various protective coatings were applied to all but the maximum tension and compression surfaces of the C-rings to prevent galvanic corrosion between the specimen and the 2024 aluminum alloy machine screw used to apply the stress. A solution of 5 weight per cent of low density polyethylene in paraffin wax formed a very durable coating on specimens tested at room temperature (Figure 1). This coating was applied by dipping at a temperature of about 120C (250F). However, because this wax formulation softens at temperatures in excess of about 60C, neoprene coatings were applied to specimens for testing at 100C.

Rather than relying on the integrity of these coatings to prevent dissimilar metal corrosion during long-term atmospheric exposures, hemispherical ceramic bushings* were placed between the aluminum stressing bolt and the C-ring (Figure 1). These bushings effectively insulated the specimens from the bolt and from other specimens when mounted on metal test frames.

In addition to tests of free-corrosion specimens, we also tested C-rings galvanically coupled

* The ceramic insulator was designed at the Aluminum Company of America Research Laboratories and can be obtained from Saxonburg Ceramics, Inc., Saxonburg, Pa., Part No. P-1861.

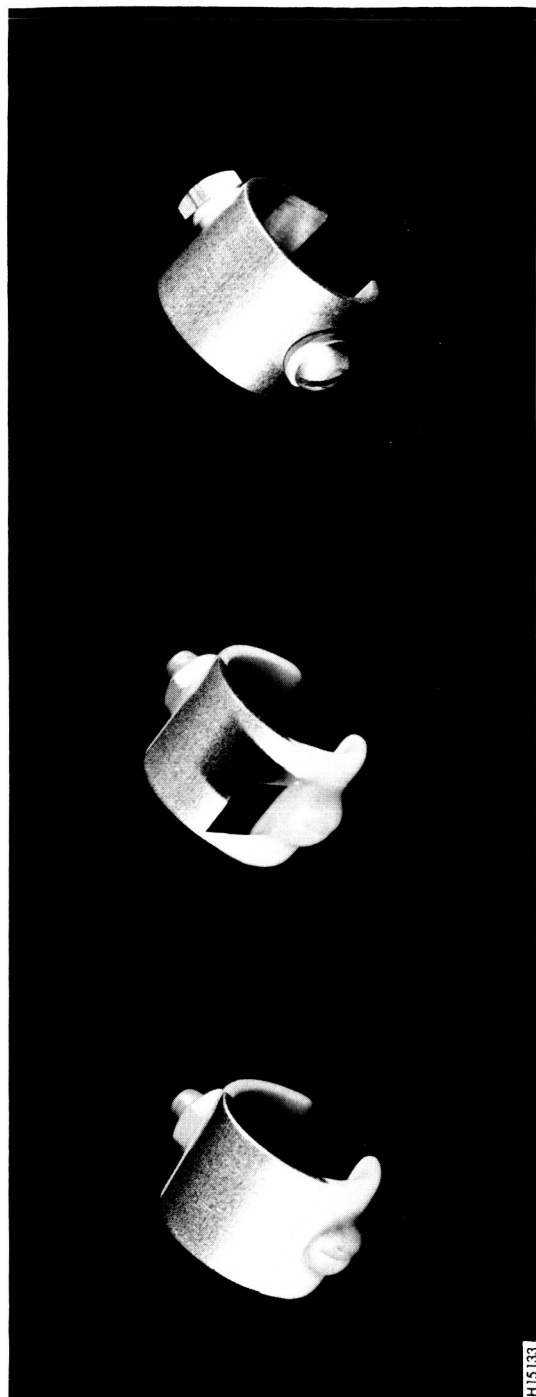


Figure 1
C-RING STRESS CORROSION TEST SPECIMENS

- Left: Free-corrosion specimen partially coated with 5% polyethylene-paraffin wax.
- Center: Specimen galvanically coupled to copper and coated as at left.
- Right: Free-corrosion specimen for atmospheric tests. Ceramic bushing isolates C-ring from the aluminum machine screw used in stressing the specimen.

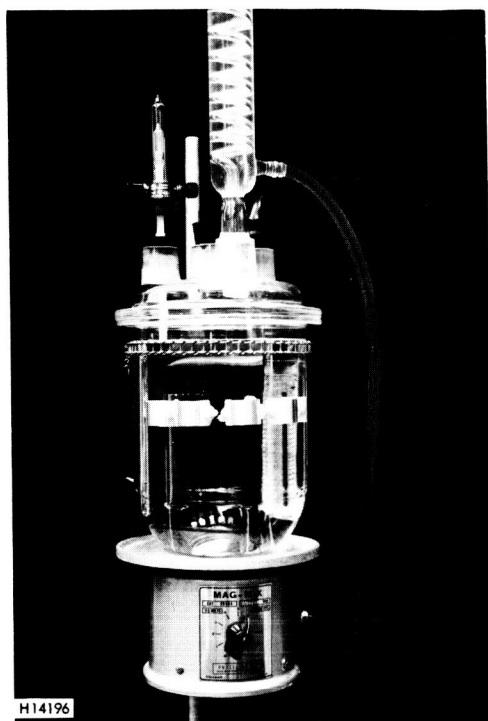


Figure 2
STRESS CORROSION TEST CELL

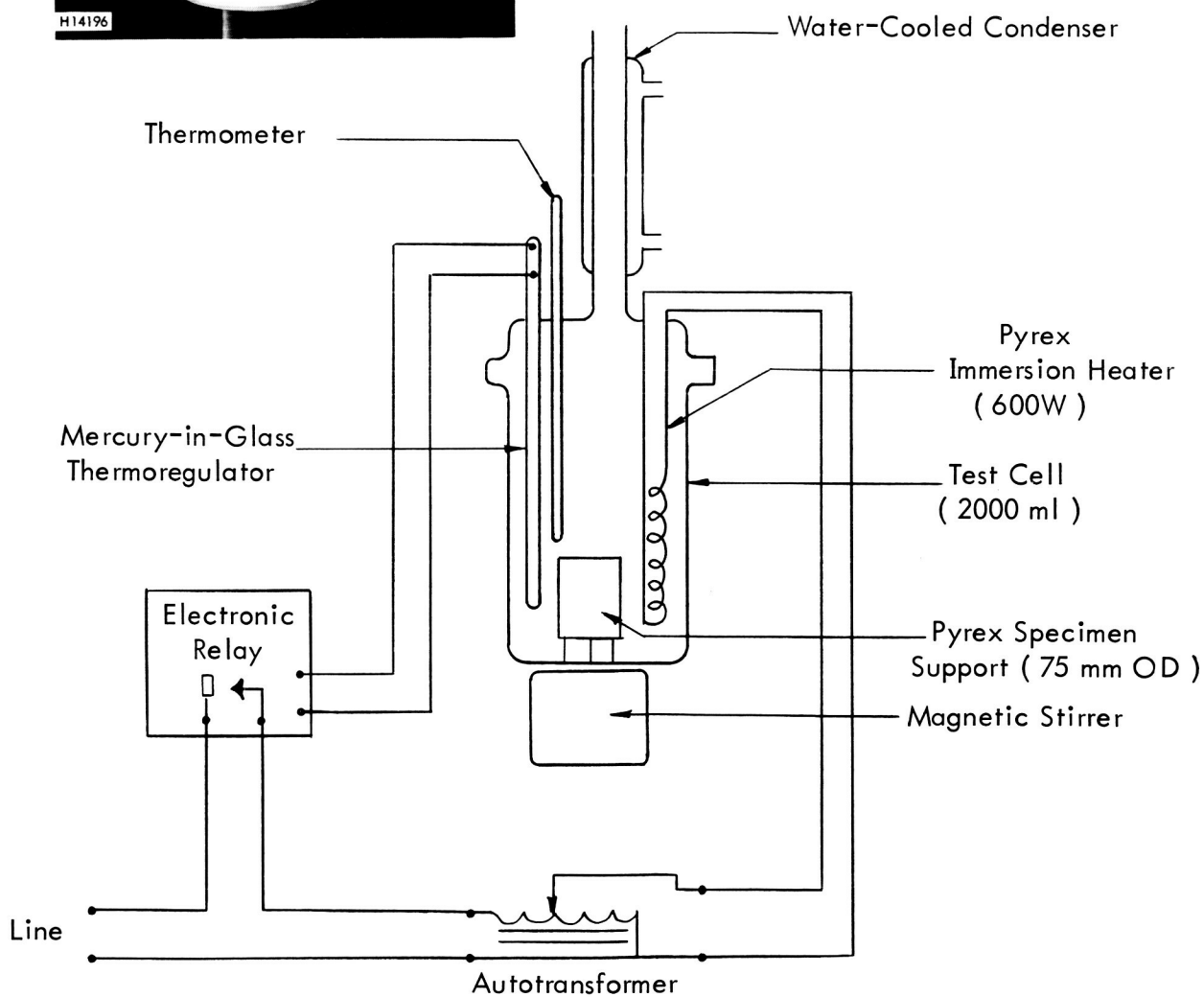


Figure 3
SCHEMATIC DIAGRAM OF APPARATUS FOR STRESS CORROSION TESTING

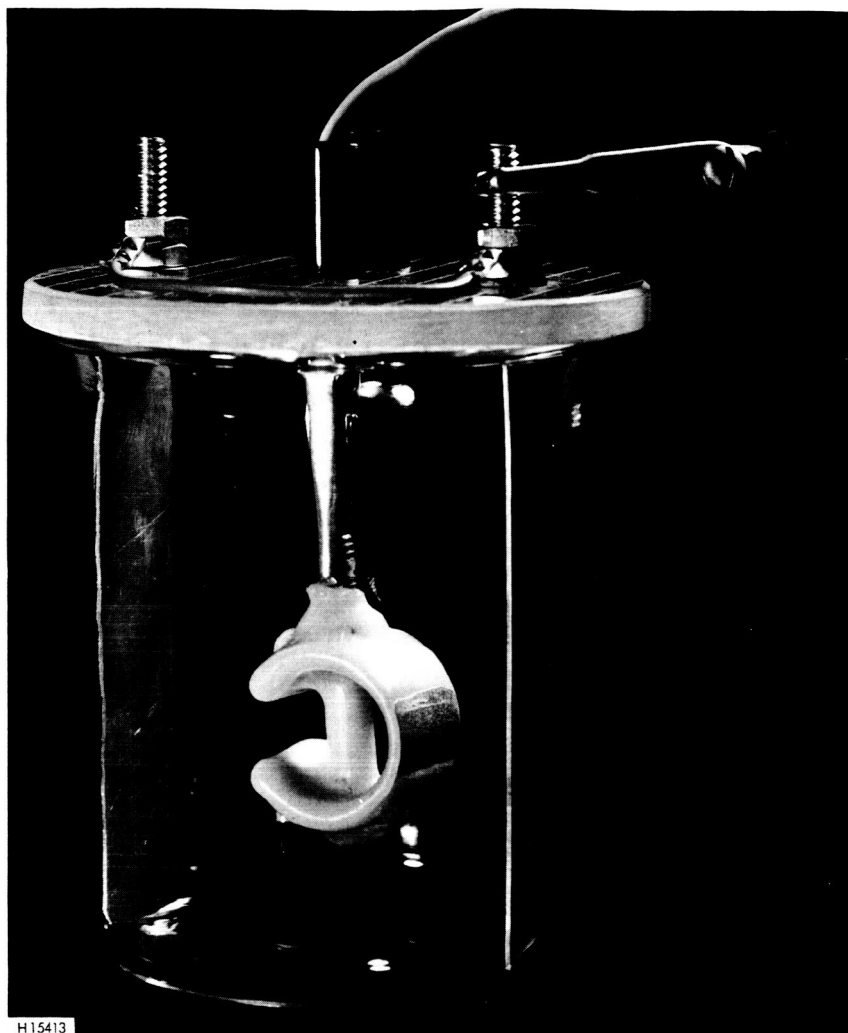
to metallic copper. High-purity copper sheet (5 mils thick) was sheared into 3/8-inch by 3/4-inch rectangular tabs. A hole was punched near one end of each tab to accommodate the 10-24 machine screw used in stressing the C-rings. The copper tab was etched in dilute nitric acid, washed in distilled water, and placed between the C-ring and the head of the stressing bolt. Each C-ring was then stressed and, as shown in Figure 1, all but the upper tension and compression surfaces of the ring and approximately 2 cm² of the copper tab were coated with a solution of 5% polyethylene in paraffin wax.

Procedures

Exploratory Stress Corrosion Tests. These tests were conducted in 4-neck 2000 ml resin reaction kettles, which were sealed and refluxed to prevent vapor loss. One of the test cells is shown in Figure 2. Figure 3 is a schematic diagram of the operating features of the apparatus. Uniform control of the temperature to $\pm 0.5^{\circ}\text{C}$ throughout each cell was maintained with a mercury-in-glass thermoregulator, electronic relay, and autotransformer control of the current input to a 600-watt Pyrex glass immersion heater. A magnetic stirrer agitated the solution.

Specimens were coated with the polyethylene-wax mixture and, while warm, affixed to the cylindrical glass support. Specimens tested at temperatures of 60°C and 100°C were coated with neoprene and held in place with Teflon hooks. This allowed continuous visual observation of the specimens through the side of the test cell. (See Figure 2 .) Evidence of cracking was detected with the aid of a 5X hand lens.

All of the exploratory stress corrosion tests were conducted by total immersion in non-aerated solutions formulated with reagent grade chemicals and 600,000 ohm-cm distilled water. The pH of these solutions was measured with a Beckman Zeromatic II pH meter. Fresh solutions were used in each test and a solution volume (ml) to specimen area (sq in.) ratio of 100: 1 or greater was maintained.



1X

Figure 4
CELL FOR INTENSIOSTATIC STRESS CORROSION TESTS

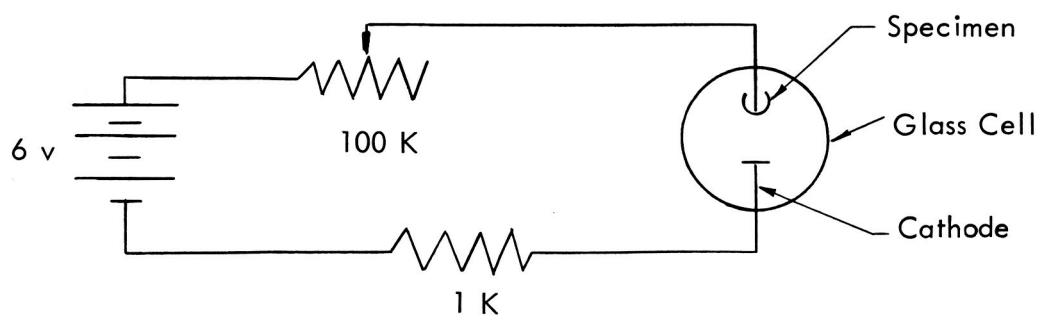


Figure 5

SCHEMATIC OF ELECTRICAL CIRCUIT FOR INTENSIOSTATIC STRESS CORROSION TESTING

Intensiostatic Tests. It is necessary to mask the C-rings used in intensiostatic tests, thereby exposing a known area of specimen surface to the electrolyte. Therefore, the stressing bolt and all surfaces except about 2 cm^2 of the tension surface of each C-ring were coated with a 5% polyethylene-95% paraffin wax mixture.

The test cell consisted of a 250-ml Pyrex beaker, with a plexiglass cover for support of the specimen and the aluminum cathodes, as shown in Figure 4. A barrel-type alligator clip attached to the stressing bolt held the C-ring in position. The alloy 1100 aluminum sheet cathodes measured 1 inch by 3 inches. These were bolted to the cover and electrically short-circuited with a wire. (This cell will also accommodate the stressing frame for the 1/8-inch diameter tensile specimens -- thus, two cathodes would be required for symmetrical distribution of current.)

The sketch, Figure 5, shows the d-c current source and the circuit for intensiostatic stress corrosion testing. A 100 K-ohm rheostat and a fixed 1 K-ohm (1%) resistor were connected in series with the test cell. The individual cells, each with a rheostat and fixed resistor, were then connected in parallel. A 6-volt lead storage battery supplied the current.

At the start of a test, the specimen and cathodes were electrically short-circuited. The current flow through the cell (corresponding to that required for a specimen current density of 50 microamperes per square centimeter) was obtained by measuring the voltage drop (IR) across the fixed 1 K-ohm resistor using a Hewlett-Packard Model 412A d-c vacuum tube voltmeter. The electrical connection between the specimen and cathodes was then opened and sufficient electrolyte (about 100 ml) was added to the cell, immersing the bare surface of the C-ring. After the cell was placed in a water bath thermostatically regulated at 30C, a final small adjustment of the current flow was made. The very small current drain on the battery required only minor current adjustments during the 7-day test period.

3.5% NaCl Alternate Immersion Tests. The test solutions were prepared with distilled water having a resistance greater than 600,000 ohm-cm and a pH of about 6.0. The purity of the NaCl used was consistent with that required by Federal Specification 151, Method 811. Solutions were replaced with freshly prepared solutions each week. Distilled water was added periodically to compensate for evaporation losses.

The alternate immersion tests were conducted under controlled conditions of temperature and humidity. The room air was conditioned at a temperature of approximately 80F and a relative humidity of 30% to 40%. The test solution was thermoregulated at a temperature of $75F \pm 2F$. The alternate immersion cycle for the 3.5% NaCl test consisted of 10 minutes' wetting and 50 minutes' drying of the specimens. This cycle was repeated hourly by pumping the solution into polyethylene test tanks with polyethylene-lined pumps. These tanks contained approximately 18 gallons of solution, which was sufficient to maintain a minimum solution volume (ml) to specimen area (sq in.) ratio of 200: 1. In addition, separate tanks were used to test copper-bearing alloys, ie, 2024, 2219, and 7075.

The time to failure of individual test specimens was determined by the first evidence of cracking noted at 15X magnification. A binocular microscope was used for this purpose.

Atmospheric Exposure Tests. The marine and industrial atmospheric exposure stress corrosion tests were initiated on 5 and 13 November, 1966, respectively. The marine exposure site is located at the Battelle Memorial Institute, North Florida Research Station, Daytona Beach, Florida, in an area approximately 500 feet from the mean tide level. Specimens were bolted to aluminum test racks which are inclined at 45° to the ground and face the ocean (easterly direction). Previous stress corrosion studies have shown that the Daytona Beach atmosphere is comparable in severity to that of the 80-foot Kure Beach, North Carolina station of the International Nickel Company. The industrial exposure site is located at an area within the McKees Rocks Industrial Park, Pittsburgh, Pa. Specimens were mounted on vertical test frames which face the northwest.

Failures in the outdoor exposure tests were detected visually (unaided) or with a 10X hand lens.

RESULTS

I EXPLORATORY STRESS CORROSION TESTS OF FREE-CORROSION SPECIMENS

pH Dependence

2024 Alloy. Previous work has shown that $\text{NaCl-H}_2\text{O}_2$ and $\text{NaCl-Na}_2\text{CrO}_4$ solutions will induce cracking in a variety of Al-Cu alloys (Refs 4 & 6). Unfortunately, the first is not stable in use -- suffering time-dependent decomposition of the peroxide -- and, therefore, subject to poor control (Refs 6 & 7). A solution containing 53 g/l NaCl + 50 g/l Na_2CrO_4 has not proved sufficiently aggressive for an accelerated test, and the results obtained are often quite erratic (Ref 3). This same solution, acidified with HCl to a $\text{pH} = 0.2$, will cause rapid cracking in susceptible tempers of 2024, but it has been reported that the rate of cracking is not much greater than that of general intergranular penetration (Ref 9). We obtained the same result, even when the chloride ion concentration of an acidified salt-dichromate solution was quite low.

For example, 2024-T4 failed in less than 10 minutes in tests where the specimens were stressed at 35 ksi and exposed to a solution containing 3 g/l NaCl + 36 g/l CrO_3 + 30 g/l $\text{K}_2\text{Cr}_2\text{O}_7^*$ at a $\text{pH} = 0.5$ and temperature of 30°C . On the other hand, 2024-T6 specimens stressed at 35 ksi failed during a 24-hour test in this same solution as a result of mixed pitting and intergranular attack on both the tension and compression surfaces of the C-rings. Obviously, stress corrosion failures and the influence of stress, if any, on intergranular attack are questionable in this case. It is interesting, however, that well-defined intergranular attack developed in 2024-T6 tested in the acidified-salt-dichromate solution at 60°C . Failures occurred in less than 20 minutes under these conditions. Yet, these failures were so rapid as to preclude adequate separation of the -T4 and -T6 products.

Recently, it was suggested that a solution containing 58.5 g/l NaCl + 50 g/l KNO_3 + 6.5 ml/l HNO_3 at a $\text{pH} = 0.85$ could be used in stress corrosion tests of various 2024 products (Ref 10). The authors stated that the accelerated test could be completed in 24 hours. We found that this solution was

* G. F. Sager, et al, (Ref 4) advocated the use of this solution in stress corrosion tests of 7075 alloy.

simply unrealistically aggressive. Thus, while -T4 specimens stressed at 35 ksi and tested at 30C failed as a result of stress corrosion cracking, -T6 specimens tested under the same conditions simply dissolved during a 5-hour exposure. Increasing the testing temperature merely served to accelerate the rate of general dissolution.

It was obvious, on the basis of these results, that highly acid media were not suited for accelerated stress corrosion tests of 2024 products. Moreover, the alkaline (ca. pH = 8.6) NaCl- Na_2CrO_4 solutions admittedly were not aggressive enough (Refs 4 & 8). Quite naturally then, electrolytes of intermediate acidity might prove more suitable. We selected salt-(di)chromate solutions for this study, on the premise that minimum general dissolution would be desirable in an accelerated test. The standard solution for these tests contained 58.5 g/l NaCl + 20 g/l $\text{K}_2\text{Cr}_2\text{O}_7$ at a pH of 3.7. The pH was changed by adding HCl or NaOH. Smaller amounts of NaCl were added to solutions at pH < 3.7 (compensating for the HCl added) so that all solutions were 1 ± 0.1 molar in chloride ion.

Table II, Part A, gives the results of short-transverse tests of 2024-T4 and -T351 C-rings stressed at 35 ksi and continuously immersed in salt-(di)chromate solutions in the range of pH = 1.0 to 7.0. The results for 2024-T4 are also graphically presented in Figure 6.

Interestingly, the rates of cracking were virtually the same at a pH of 1 or 7. On the other hand, a delay in cracking occurred at intermediate values of pH, with the maximum failure times for both the -T4 and -T351 items occurring at approximately pH = 3.0. An explanation for this unusual behavior, based on the competing rates of general corrosion and stress-accelerated intergranular attack, was considered in our 5th Progress Report (15 August, 1966). For the purposes of this discussion, however, it is sufficient to note that a neutral pH solution appeared most suited for use in an accelerated test of 2024-T4 and -T351.

On this assumption, we proceeded with tests of 2024-T6. The standard (1.0 M Cl^-) - (di)chromate solution at a pH = 7.0 produced failures in -T6 specimens stressed at 45 and 55 ksi, but

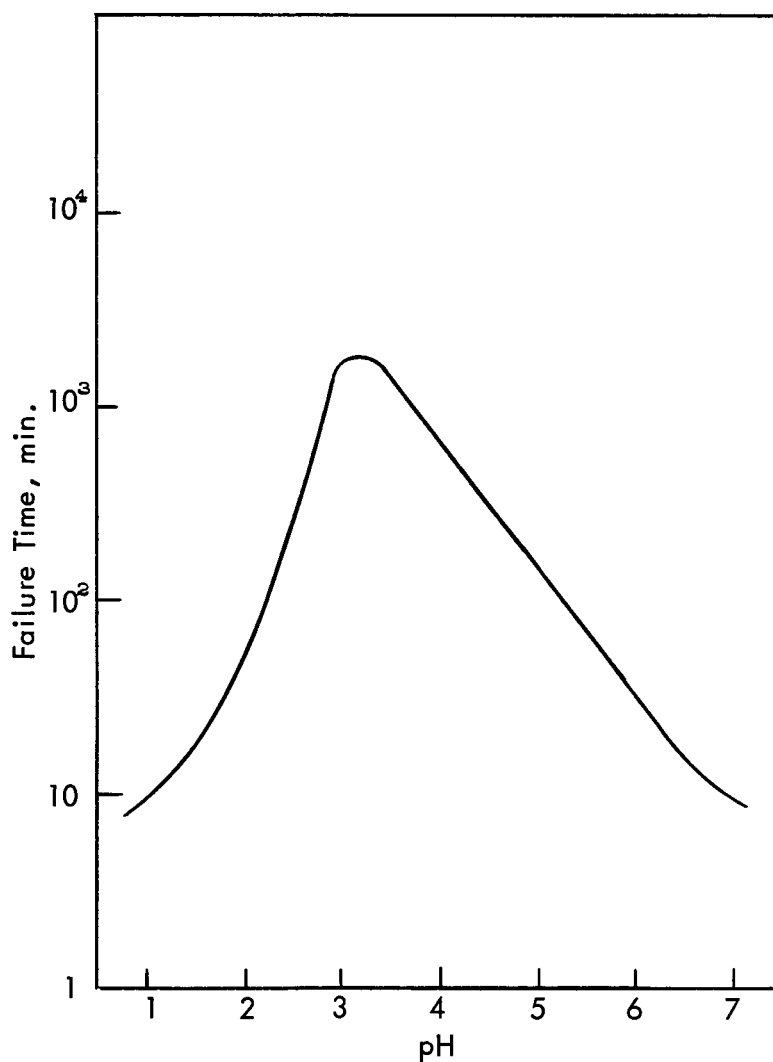


Figure 6

EFFECT OF pH ON THE STRESS CORROSION RESISTANCE
OF 2024-T4 IN SALT-(DI) CHROMATE SOLUTIONS AT 30C

not in specimens stressed at 35 ksi. See Table II, Part A. While there was adequate separation of the -T4 and -T6 items, the results for -T6 were somewhat erratic. That is, one specimen stressed at 45 ksi failed in two days, two specimens stressed at 55 ksi failed in one to two days, while the remaining specimens did not fail in more than 14 days of testing.

More rapid failures and less erratic results for 2024-T6 were obtained in the neutral salt-(di)chromate solutions by increasing the chloride ion concentration to 2.0 M. These data are given in Table II, Part B. While this solution may have been too aggressive, since failures eventually occurred in the -T6 specimens stressed at 35 ksi, the failure times were dependent upon the level of stress. Tests of -T6 specimens in solutions 5.0 M in chloride ion showed that this electrolyte was too aggressive (Table II, Part C).

At this point, we concluded that a neutral salt-(di)chromate solution containing between 6% and 12% NaCl could be used in an accelerated stress corrosion test of 2024 products.

2219 Alloy. We anticipated that the performance of susceptible tempers of 2219 exposed to salt-(di)chromate solutions would be analogous to that of 2024-T4 and -T351. For example, 2219-T42 short-transverse C-rings were stressed at 20 ksi and exposed to salt (1.0 M Cl^-) -(di)chromate solutions at various pH values between 1 and 7 and at a temperature of 30C. As shown in Table III and Figure 7, maximum resistance to stress corrosion occurred at a pH between 2.0 and 3.0, whereas failures occurred in a matter of one to two hours at a pH of 1.0 and 7.0. In fact, these data virtually paralleled those for 2024-T4 and -T351, except that the failure times for 2219 were somewhat longer at each pH value.

The delay in cracking of 2219-T42 in the range of pH = 2 to 3 is similar to that reported by Lifka, et al (see Table 10, Ref 8) for 2219-T37 tested by alternate immersion in acidified salt-di-chromate solutions at room temperature. In these tests, increasing longer times to failure were observed as the pH of the solution was increased in the range pH 1.0 to pH = 2.3 and 2.7. Apparently, this behavior is characteristic of alloys containing large amounts of copper, whether they are tested by

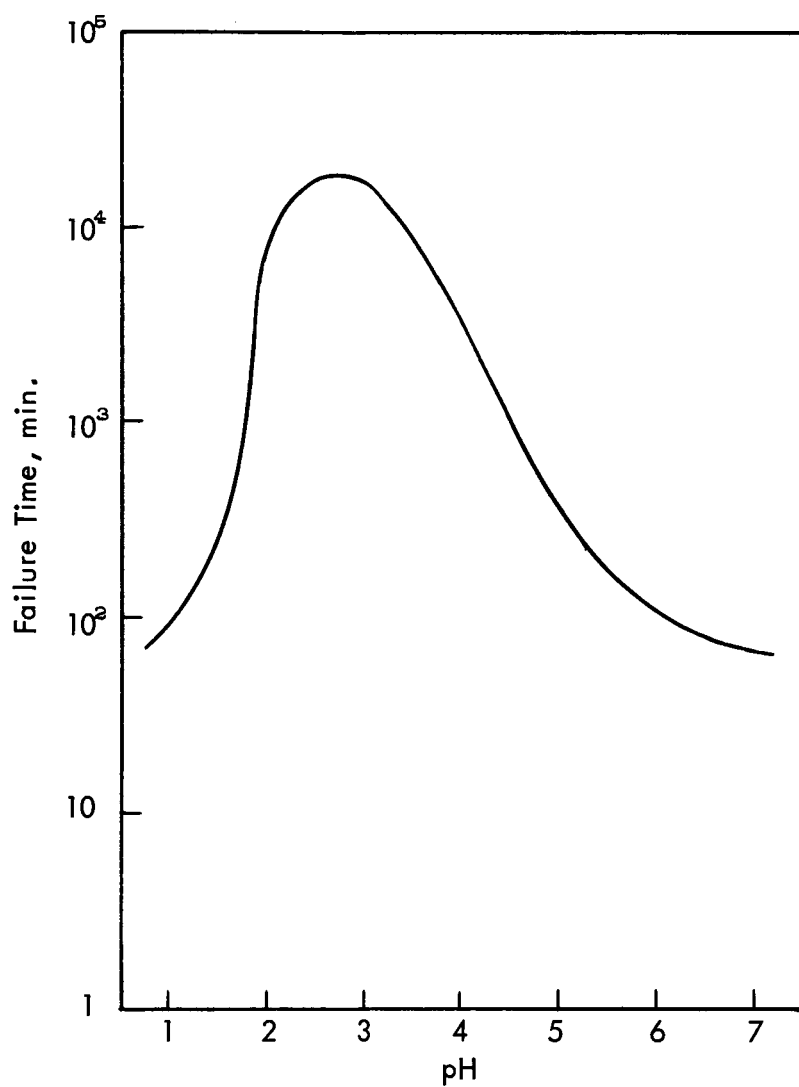


Figure 7
EFFECT OF pH ON STRESS CORROSION OF
2219-T42 IN SALT-(DI) CHROMATE SOLUTIONS AT 30C

alternate immersion or by continuous immersion in acidified salt-(di)chromate solutions. The question remained -- would Al-Zn-Mg-Cu (7075) or Al-Zn-Mg (7039) alloys behave similarly ?

7075 Alloy. Comparative test data for 7075-T651 C-rings stressed at 45 ksi are given in Table IV and Figure 8. In this instance, maximum resistance to short-transverse stress corrosion occurred in the range of pH = 4.5 to 6.5. The inflections in the pH-failure time curve (Figure 8) at approximately pH = 4.5 and 6.5 are not easily explained. We expected that decreasing hydrogen ion concentration (increasing pH) would lead to increasingly longer times to failure. However, solutions in this range of pH contain partially dissociated bichromic acid. Therefore, the buffering capacity (total acidity) of these media would be greater than that of solutions containing a strong totally dissociated acid. Consequently, it does not seem reasonable that increasing pH, per se, would delay cracking, ie, depolarization or removal of discharged hydrogen may be more important. We made no effort to aerate these media, and therefore, varying oxygen concentration may have contributed to these anomalous results. Certainly, this may explain why, in several repeat tests in neutral solutions, rapid propagation of cracks did not always occur. A few specimens would exhibit rapid cracking in an hour or less, whereas others remained intact for days. Of course, we did not anticipate complete separation of the C-rings. But, in these tests of 7075-T651, crack propagation was much more erratic than would be expected for specimens stressed at 45,000 psi. Obviously, more reproducible response would be required if the neutral salt-(di)chromate solutions were to be applicable in accelerated stress corrosion tests of 7075 products.

7039 Alloy. Unlike the results with alloys 2024, 2219 and 7075, the stress corrosion resistance of 7039-T64 specimens stressed at 35 ksi and exposed to the salt (1.0 M Cl^-)-(di)chromate solutions simply increased with pH. See Table V and Figure 9. The similarity in the stress corrosion performance of both lots of 7039 at pH = 1.0 would preclude adequate separation of these materials. And, while there was greater separation in their performance with increasing pH, no failures occurred in lot 742101 during a 14-day exposure at a pH = 7.0. This suggested that, like 7075, more aggressive conditions would be required for an accelerated test of 7039.

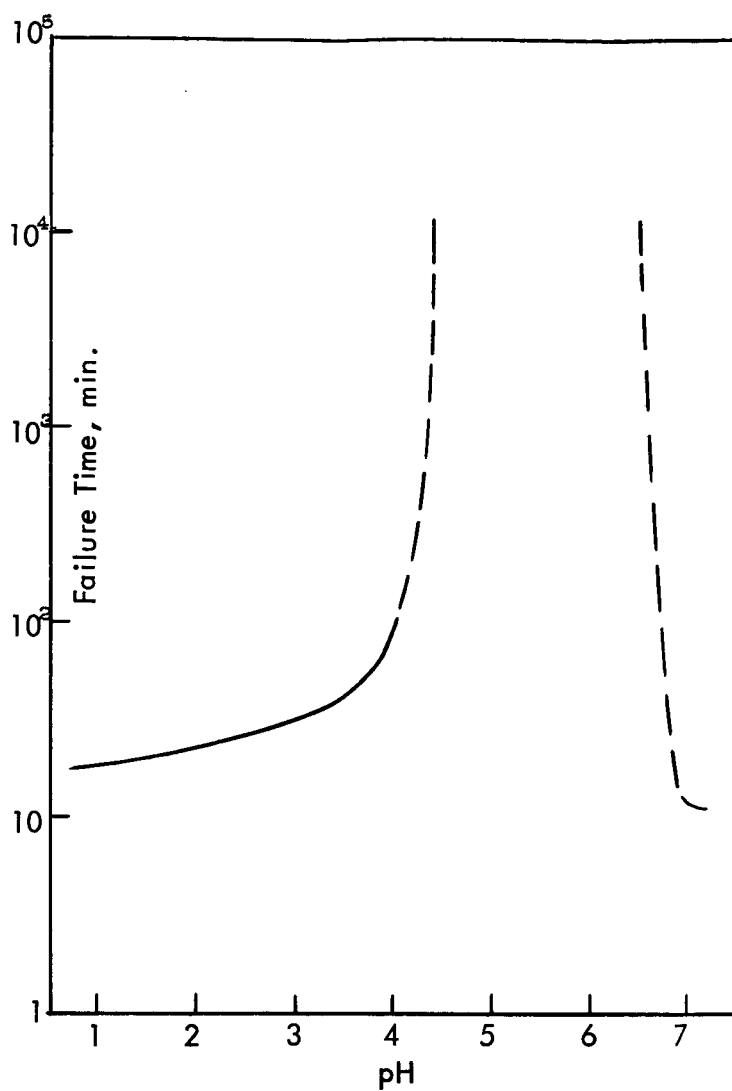
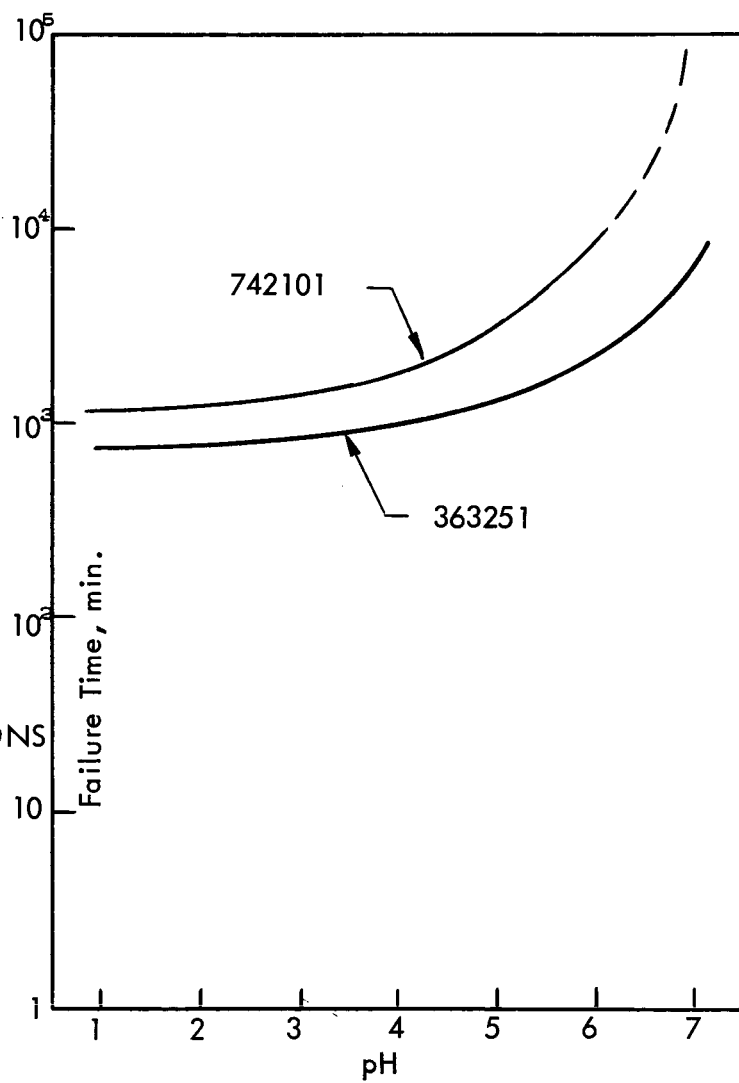


Figure 8

EFFECT OF pH ON STRESS CORROSION OF
7075-T651 IN SALT-(DI) CHROMATE SOLUTIONS
AT 30C

Figure 9

EFFECT OF pH ON STRESS CORROSION OF
7039-T64 IN SALT-(DI) CHROMATE SOLUTIONS
AT 30C



Temperature Dependence

Previous studies, conducted in our laboratories (Ref 11) and elsewhere (Ref 12), demonstrated that continuous immersion in simple NaCl solutions was quite effective in promoting rapid stress corrosion failures in Al-Zn-Mg alloys. Cracking was observed in minutes or days, dependent upon the degree of susceptibility, level of applied stress, and test temperature. As shown in Table VI, increasing the temperature of a 1 molar NaCl solution from 30C to 100C resulted in almost a 1000-fold increase in the rate of cracking for all of the 7039 items tested. Moreover, in each case, we obtained a linear relationship in plots of the log failure times versus the inverse of the absolute test temperature-- leaving little doubt that stress corrosion of 7039 alloy was thermally activated (Ref 11).

Recently, Lifka and Sprowls (Ref 2) demonstrated that a boiling 6% (ca. 1 M) NaCl solution was effective in accelerating short-transverse stress corrosion cracking of 7079-T6. However, they correctly noted that continuous immersion in boiling NaCl solutions was not effective on Al-Cu, Al-Mg or Al-Zn-Mg alloys containing more than about 1% Cu. We also recognized that, except for Al-Mg and Al-Zn-Mg alloys, continuous immersion in simple salt solutions at room temperature was ineffective in tests of copper-bearing alloys such as 2024, 2219 and 7075. Yet, as we have shown, these alloys can be tested by continuous immersion in salt-(di)chromate solution. As such, it was likely that the temperature of these solutions would determine in large measure if, or how rapidly, stress corrosion cracking would occur.

2024 Alloy. There was no significant increase in the rate of cracking in 2024-T4 specimens stressed at 35 ksi and tested between 0C and 100C in salt (1 M Cl^-)-(di)chromate solutions at a pH of 1.0 (Table VII). Of course, we did not anticipate using this solution in a practical stress corrosion test, but these results confirmed Nichols and Rostoker's observation (Refs 9 & 13) that stress corrosion of 2024 in salt-(di)chromate solutions at pH ≥ 1 was independent of temperature. Significantly, however, in the standard salt-(di)chromate solution at pH = 3.7, approximately a 1000-fold increase

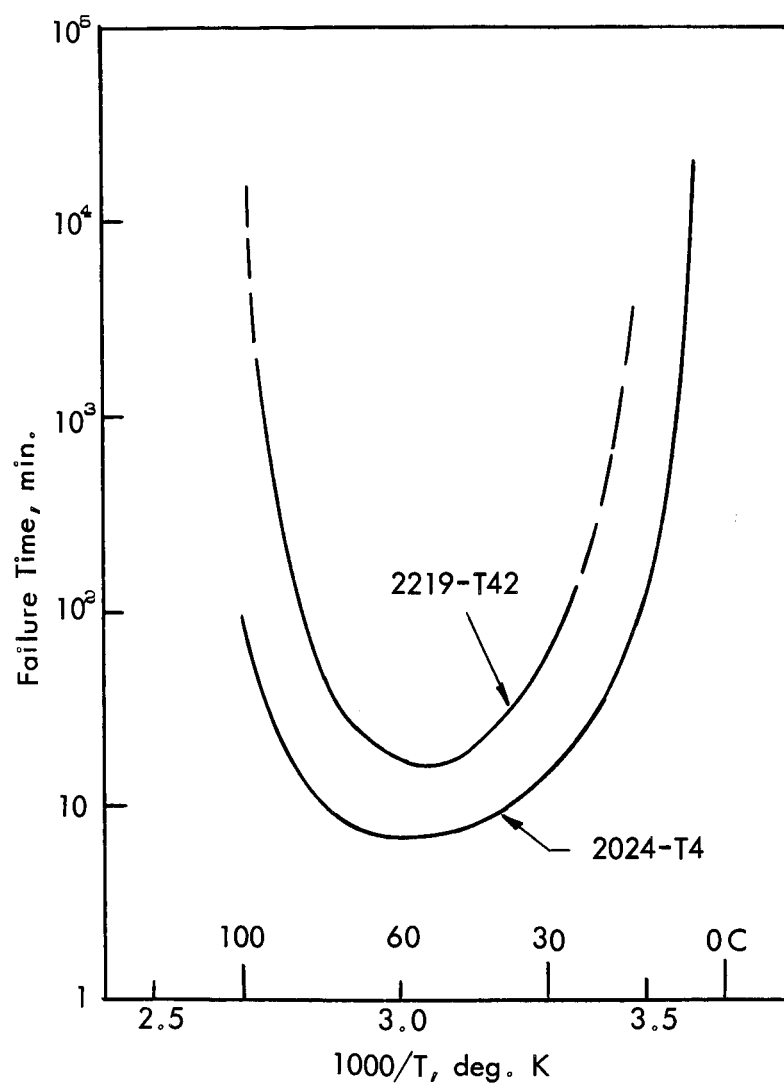


Figure 10

EFFECT OF TEMPERATURE ON STRESS CORROSION OF 2024-T4
AND 2219-T42 IN NEUTRAL SALT-(DI) CHROMATE SOLUTIONS

in the rate of cracking occurred between 0C and 100C. Moreover, failures were observed in about the same time at pH = 3.7 and 100C as at pH = 7 and 30C or 60C, suggesting for the moment that the pH = 3.7 solution might be suitable for use in an accelerated test.

Our interest, of course, was directed toward obtaining more aggressive test conditions using neutral solutions. Since the rate of cracking in 2024-T4 was quite rapid at pH = 7.0 and 30C, we did not expect a marked increase in this rate with increasing temperature above 30C, ie, the behavior of 2024-T4 would be similar to that observed at pH = 1.0. Certainly, this was true at a temperature of 60C. On the other hand, cracking was delayed at 100C and pH = 6.9, giving the u-shaped plot of the log failure times versus the inverse of the absolute test temperature as shown in Figure 10.

No well-defined region of linearity occurred, as would be typical for a thermally activated process. The plot (Figure 10) merely suggested that (at least) two competing reactions determine the over-all rate of stress corrosion cracking in 2024-T4. As a result, we would not expect that stress corrosion would be thermally activated over a very wide range of temperatures, nor would the range of thermal activation be the same for different alloys.

2219 Alloy. We had no reason to believe that the elevated temperature performance of 2219-T42 and 2024-T4 would differ in acidified salt-(di)chromate solutions. And, again, we were more interested in the behavior of these products in a neutral solution. As shown in Table VII and Figure 10, the rate of cracking at 60C and pH = 7.0 increased slightly over that at 30C. However, as in the tests of 2024, cracking in 2219-T42 was delayed at a temperature of 100C. In fact, no failures were detected during a 3-day test at this temperature, suggesting that 2024 was more susceptible to intergranular attack than was 2219. These data also suggest that, for the purpose of developing a rapid test based on the use of neutral salt-(di)chromate solutions, temperatures much in excess of 60C should be avoided.

7075 Alloy. The stress corrosion performance of 7075-T651 specimens stressed at 45 ksi and continuously immersed in salt (1.0 M Cl⁻)-(di)chromate solutions at pH = 1.0 and 3.7 was in marked contrast to that of the 2024-T4. As shown in Table VIII, increasing the temperature (above 30C)

virtually eliminated cracking. The two failures of 7075-T651 specimens exposed at pH = 3.7 and 100C appeared anomalous, since a retest under these same conditions failed to produce stress corrosion cracking.

As expected, decreasing the temperature below 30C lengthened the time to failure for -T651 specimens. The net result was the generation of the u-shaped time temperature curves shown in Figure 11. It is not impossible that stress corrosion is thermally activated below about 30C, but we must invoke the argument that other processes (possibly general corrosion) interfere with cracking at higher temperatures.

While slightly faster rates of cracking occurred at pH = 1.0, as compared with pH = 3.7, the minimum time to failure occurred at 30C in both media. Therefore, we discounted our previous suggestion that a boiling solution at pH = 3.7 could be used in an accelerated test for all aluminum alloys. Moreover, increasing the temperature of the neutral salt-(di)chromate solutions to 60C or 100C also proved unsuccessful as a means of inducing more rapid cracking in 7075-T651. As such, we were faced with the problem of finding alternate methods for conducting accelerated stress corrosion tests of 7075 products.

7039 Alloy. Unlike the results with 7075-T651, increasing the temperature of the salt-(di)chromate solutions at either pH = 1, 3.7 or 7 resulted in more rapid failures in 7039-T64 (Table IX). As shown in Figure 12, the linear relationship between the logarithm of the time to failure and the inverse of the absolute test temperature demonstrates that stress corrosion of 7039 is a thermally activated process.

Increasing hydrogen ion concentration resulted in somewhat faster rates of cracking in 7039 and the pH = 1.0 solution was quite effective in causing stress corrosion -- even at 0C. Previous experience had shown that protracted exposures to neutral salt solutions at a temperature of 0C would not result in failures. Apparently, then, the effect of low pH was to widen the range of temperature over which stress corrosion can occur in 7039.

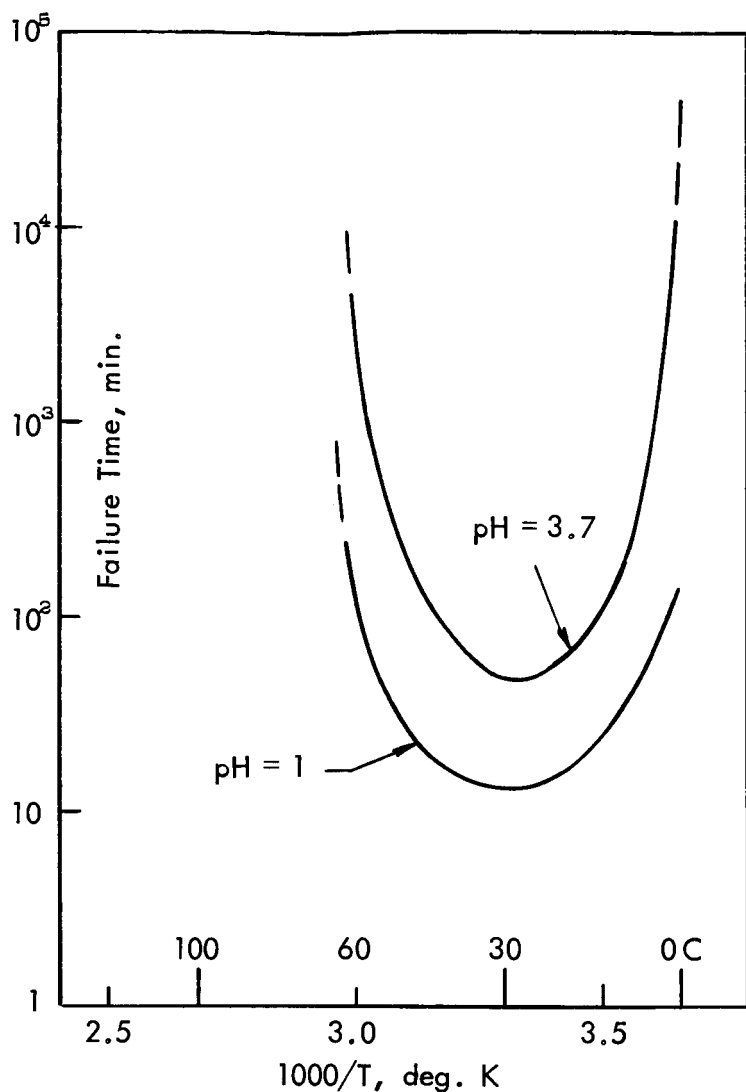
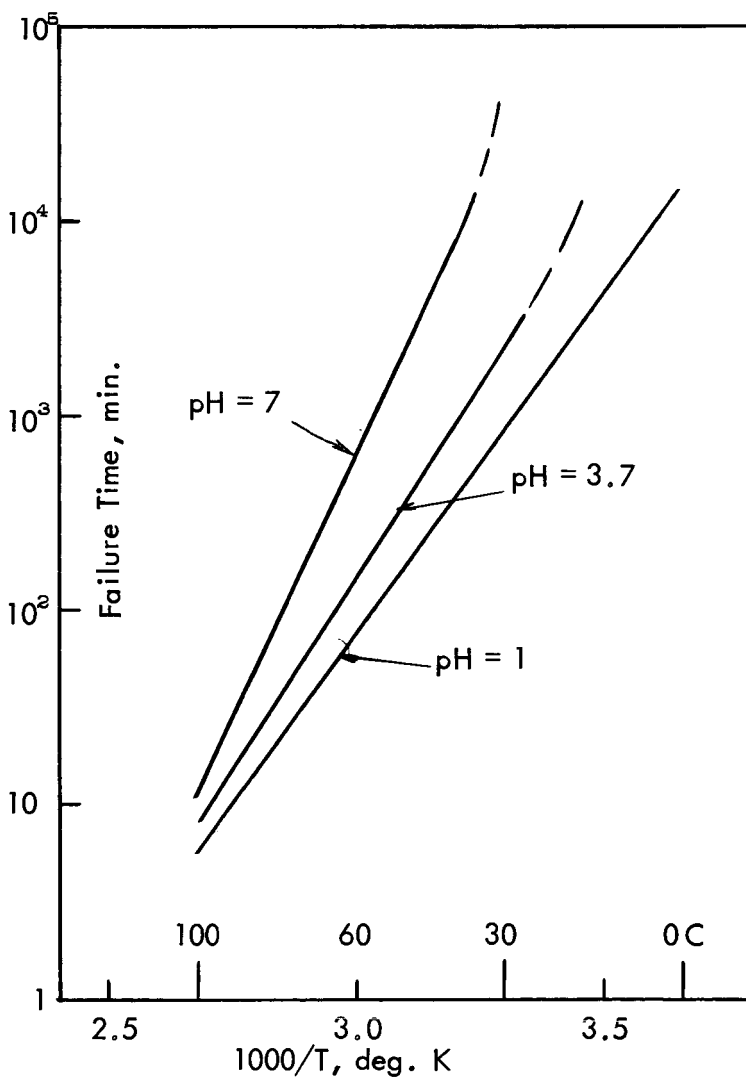


Figure 11

EFFECT OF TEMPERATURE ON STRESS CORROSION OF 7075-T651 IN SALT-(DI) CHROMATE SOLUTIONS AT pH = 1 AND pH = 3.7

Figure 12

EFFECT OF TEMPERATURE ON STRESS CORROSION OF 7039-T64 (742101) IN SALT-(DI) CHROMATE SOLUTIONS AT pH = 1, 3.7 AND 7



II STRESS CORROSION TESTS OF GALVANIC COUPLES

The rate of corrosion on aluminum alloys in electrolyte solutions can be increased by applying a suitable driving emf between the alloy (anode) and an auxiliary electrode (cathode). The total anodic current determines the rate of aluminum corrosion. Depending on whether the potential or current is controlled, the test is described as potentiostatic or intensiostatic, respectively. Several workers have discussed (Refs 14 through 18) these methods of controlling tests of the susceptibility to intergranular corrosion and stress corrosion cracking of aluminum alloys. The intensiostatic tests are easily applied in practice and require only a simple battery and resistance circuit. However, a far simpler approach would involve coupling the test specimen to a more noble metal or alloy; in effect, defining both the driving emf and current for intergranular attack or stress corrosion cracking.

We recognized that metallic copper precipitates (galvanic displacement) on the surfaces of copper-bearing aluminum alloys tested by alternate immersion in 3.5% NaCl solution. In these instances, it can reasonably be assumed that copper acts as an effective cathode during stress corrosion cracking. If this assumption is correct, we would expect that more aggressive test conditions [in the neutral salt -(di)chromate solutions] could be achieved by physically coupling metallic copper to the test specimens. Certainly, this approach was attractive from the standpoint that the source of driving emf for stress corrosion would be defined.

The short-transverse stress corrosion performance of 2024, 2219, 7075 and 7039 C-rings galvanically coupled to metallic copper and tested in a neutral 5% NaCl-2% $K_2Cr_2O_7$ solution at 30C is recorded in Table X. As expected, rapid failures occurred in the 2024-T4, -T351, 2219-T42, -T351 and -T37 specimens. However, the failure times were no shorter than that for freely corroding specimens tested under comparable conditions, even though there were greater numbers of cracks in the galvanically coupled specimens. It is possible that individual cracks can only propagate at some finite rate, dependent upon the current density at the crack tip. The maximum or limiting current density might be achieved at one or more cracks under conditions of free corrosion (possibly a small total current, but few cracks).

In this event, increasing the total current on the specimen (galvanically) would not accelerate crack propagation. Rather, it might be expected that greater numbers of potential crack sites would develop into active propagating cracks -- as observed experimentally.

Previously, when cracking occurred in freely corroding 7075-T651 exposed to neutral salt-(di)chromate solutions at a stress of 45,000 psi, rapid propagation of cracks did not always follow. Moreover, in repeated tests under identical exposure conditions, some of the 7075-T651 specimens failed to show any evidence of intergranular attack. However, under conditions of imposed galvanic attack, failure of 7075-T651 stressed at 45,000 psi were extremely rapid. In some instances, virtually complete separation of the C-ring specimen occurred in 90 to 120 minutes.

Stress corrosion cracking of 7039-T64, freely corroding in a neutral salt-(di)chromate solution at 30C, was previously observed only in the most susceptible lot (363251) tested. On the other hand, relatively rapid failures occurred in all of the 7039 items when the C-rings were galvanically coupled to copper. As shown in Table X, the more susceptible lot (363251) of 7039-T64 exhibited shorter times to failure. However, a ranking of the remaining items would be more difficult. Possibly, a comparison at lower stresses or on the basis of threshold stresses would have been more meaningful.

The more stress corrosion resistant tempers of 2024, 2219 and 7075 performed as expected. It is significant that only one failure was observed in the Cu:2024-T6 couples stressed at 35,000 psi. Yet, at higher stresses, all of the 2024-T6 failed during the 7-day test. Previous work indicated that an approach to a threshold stress between 35,000 and 45,000 psi could be obtained for freely corroding specimens tested in a 6% NaCl-(di)chromate solution. This level was achieved, however, at the cost of excessive scatter in the failure times of specimens stressed at 55,000 psi. Increasing the chloride ion concentration (to about 12% NaCl) eliminated this erratic behavior; however, failures then occurred at 35,000 psi. Thus, we reasoned that it might be possible to obtain more definitive results by testing galvanically coupled specimens in salt-(di)chromate solutions containing less than 5% NaCl, viz, a 3% NaCl solution.

There is, however, a major drawback to this test method. The magnitude of the galvanic currents was sensitive to solution agitation (at the copper) and, unless special precautions were taken, there was no assurance that the same rate of agitation would be maintained at all specimens. Thus, while this may duplicate the conditions existing during free-corrosion of aluminum alloys, when local cathodic sites operate at the bulk solution-specimen interface, this condition does not lend itself to close control of the rates of general corrosion or stress corrosion cracking. On the other hand, impressed currents obtained from battery sources are not necessarily subject to this limitation. If the total anodic current is determined by the potential drop (resistance) of the external battery circuit, the corresponding corrosion rate will be subject to improved control*.

III INTENSIOSTATIC STRESS CORROSION TESTS

Measurements of the zero-resistance galvanic currents ranged from 40 microamperes in tests of 2024-T4 to 130 microamperes in tests of 7039-T64 when specimens were coupled to 2 cm² of copper and exposed to neutral salt-(di)chromate solutions at 30C. For practical reasons, we selected an anodic current density of 50 microamperes/cm² for use in all intensiostatic tests. Table XI gives the results of a 7-day intensiostatic stress corrosion test of 2024, 2219 and 7075 short-transverse C-rings exposed to a neutral 3% NaCl-2% K₂Cr₂O₇ solution at 30C. As we expected, the highly stressed susceptible products, such as 2024-T351, 2219-T351 and 7075-T651, etc, failed within an hour or less. On the other hand, the more stress corrosion resistant tempers of these same alloys withstood the attack for days or, at applied stresses near their short-transverse threshold stress, exhibited freedom from cracking during the 7-day test period.

Thus, while this intensiostatic test shows promise as a method of separating stress corrosion resistant and highly susceptible tempers of alloys 2024, 2219 and 7075, this is not sufficient for qualification of a new test method. At the time of this writing, we had not tested the 7039 products,

* The external resistance of the battery circuits used in intensiostatic corrosion tests is made so large that the potential drop in the electrolyte is negligible by comparison.

although the results should be comparable to those obtained when the specimens were galvanically coupled to copper. Moreover, we will also require alternate immersion and intensiostatic tests of 2024-T4, -T351; 2219-T42, -T351, -T37; and 7075-T651 plate samples which have been artificially aged to exhibit borderline resistance to short-transverse stress corrosion. Only then will we have assurance that a 7-day intensiostatic test is sufficient to qualify 2024-T6, -T851; 2219-T62, -T851, -T87; and 7075-T7351 as stress corrosion resistant products.

IV 3.5% NaCl ALTERNATE IMMERSION TESTS

Table XII gives the results of 90 days' alternate immersion testing of 2024 and 7075, 30 days' testing of 2219, and the results obtained to date for alloy 7039. The 7039-T64 (363251) has been in test 184 days, whereas, the remaining 7039 items have been in test 287 days, as of March 1.

Rapid short-transverse stress corrosion was observed in 2024-T4, -T351 and 7075-T651 specimens at applied stresses of 10 ksi or greater. As expected, the frequency of failures decreased and the times to failure increased with decreasing load, suggesting an approach to an endurance of threshold stress of between 5 ksi and 10 ksi. However, it was found that specimens stressed at 5 ksi and examined after 30, 60 and 90 days' exposure exhibited marked intergranular attack on both the compression and tension surfaces of the C-rings. Thus, while it was considered instructive to continue these tests for 90 days, a 30-day test of 2024-T4, -T351 and 7075-T651 would be adequate in most instances.

With the exception of deep pitting and general surface attack, failures in 2024-T6, -T851 and 7075-T7351 occurred only at high sustained stresses during relatively longer periods of exposure. No failures were observed in 2024-T6 and -T851 specimens stressed at 30 and 35 ksi or in 7075-T7351 specimens stressed at 45 ksi. Failures in 7075-T7351 specimens stressed at 50 ksi were detected solely on the basis of microscopic examination following termination of the 90-day test.

The 2219-T42, -T351 and -T37 were susceptible to short-transverse cracking at applied stresses of 10 ksi or greater. However, as is characteristic of the alternate immersion testing of copper-bearing aluminum alloys, if failures were not detected during the first week or so of testing, general

surface corrosion greatly impaired visual detection of fine cracks. As a result, the majority of failures in the -T42, -T351 and -T37 specimens initially stressed at 10 ksi were detected solely on the basis of metallographic examination of specimen cross sections. Failures in specimens stressed at 5 ksi were questionable, since intergranular attack of both the compression and tension surfaces of the C-rings was so extensive as to preclude separation of the effects of stress corrosion and general intergranular corrosion.

By comparison, the stress corrosion performance of 2219-T62, -T851 and -T87 was much better. No failures were detected visually or under low-power microscopic examination during the 30-day exposure. Therefore, all specimens were sectioned upon termination of the test and examined metallographically for evidence of intergranular attack. Again, no failures were observed in the -T62 specimens. One failure was detected in the group of -T851 specimens stressed at 90% of the long-transverse yield strength. While the frequency of failures in the -T87 specimens was somewhat higher, with failures occurring at applied stresses equal to or greater than 75% of the yield strength, these failures occurred only at the relatively high stresses of at least 45 ksi.

The 7039, being a copper-free alloy, exhibited excellent resistance to general corrosion, but was susceptible to short-transverse stress corrosion. As shown in Table XII, failures were observed in 7039-T64, lot 363251 specimens stressed at 15 ksi and above, and in specimens of -T64, lot 742101, stressed at 10 ksi and above. The performance of 7039-T61 and -T6E132 appeared better in these alternate immersion tests. A singular failure occurred in the -T6E132 at 20 ksi, otherwise, failures have not occurred in the 7039-T61 at 20 ksi or less in more than 287 days' testing.

V MARINE AND INDUSTRIAL ATMOSPHERE TESTS

Alloys 2024, 2219, 7075 and 7039 (five specimens for each variable of stress and temper as in the alternate immersion tests) have been in test 117 days at Daytona Beach, Florida. As shown in Table XIII, failures in the marine environment have been observed only in the more susceptible products and tempers, viz, 2024-T4, -T351, 2219-T42, -T351, -T37, 7075-T651 and 7039 (all tempers).

Failures have not been observed in 2024-T6 or -T851 at applied stresses of 50 ksi or below, in 2219-T62, -T851 or -T87 at 35, 50 and 55 ksi or less, respectively, nor in 7075-T7351 at stresses of 50 ksi or less.

Alloys 2024, 2219, 7075 and 7039 have been in test for 109 days at Pittsburgh Pa. As shown in Table XIV, failures have been observed only in 7075-T651 and the 7039. There is little doubt that the alternate immersion test and marine exposure were more effective than the industrial exposure in promoting stress corrosion of 7075-T651. On the other hand, the performance of the 7039 in these tests was quite different. Consequently, a comparison of the effectiveness of alternate immersion testing over marine and industrial atmosphere tests should prove interesting. In Figure 13, we have plotted the frequency of failures observed in these tests of 7039 versus the applied tensile stress. Excepting the -T6E132 temper, the marine and industrial exposures were equally effective in causing stress corrosion in 7039. But, more important, they were consistently more effective than alternate immersion testing. These results are particularly striking when one considers that the period in alternate immersion testing was approximately 80 or 180 days longer than that in either of the service environments. As such, the alternate immersion test can hardly be regarded as an accelerated test for 7039*.

The Daytona Beach marine atmosphere was very effective in causing short-transverse stress corrosion of 7039 (all tempers), 7075-T651 and susceptible tempers of 2024 and 2219. Yet, little doubt exists that 7039 was more prone to failure in the Pittsburgh environment than 7075-T651 or, in particular, susceptible tempers of 2024 and 2219. Obviously then, in the development of an accelerated stress corrosion test, consideration must be given to the fact that no one test, laboratory or service environment, will rank all alloys or heat treatments in the same order of susceptibility. Possibly, the best that can be achieved (in laboratory tests) is a demonstration of susceptibility or resistance to stress corrosion cracking in much shorter periods of exposure.

* Lifka and Sprowls (Ref 2) have also shown that at low stress levels 7039 is more likely to fail in atmospheric tests than in a 3.5% NaCl alternate immersion test.

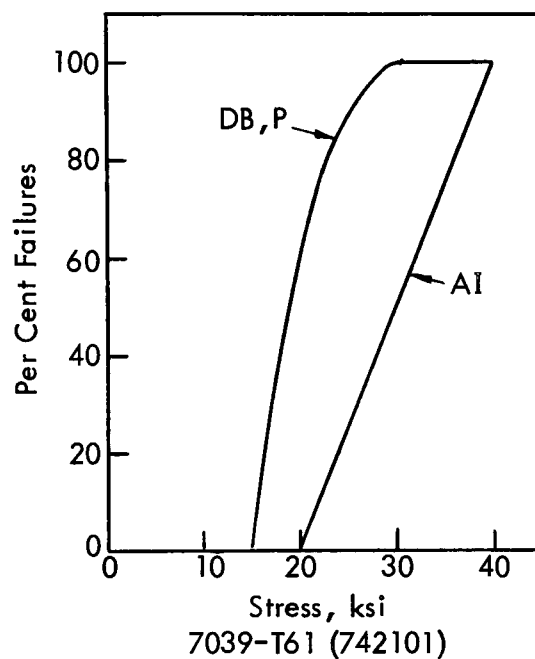
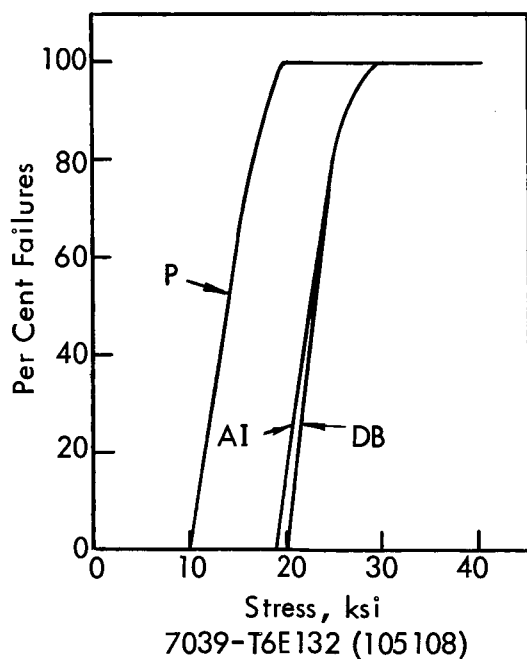
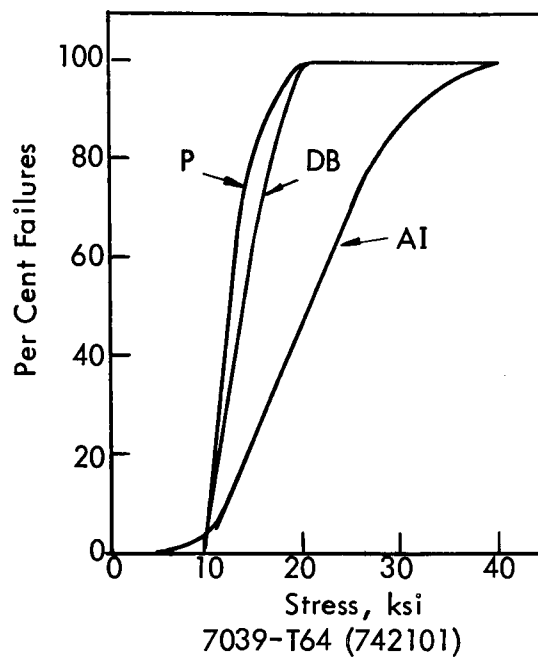
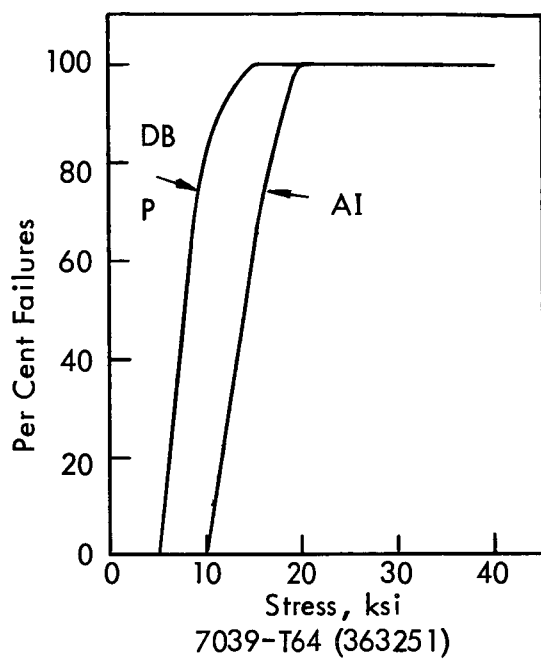


Figure 13

EFFECT OF ENVIRONMENT ON THE SHORT-TRANSVERSE STRESS CORROSION
RESISTANCE OF ALLOY 7039

AI = Alternate Immersion in 3.5% NaCl, Lot 363251 in test 184 d, all others, 287 d;
DB = Daytona Beach, Florida (marine) 117 d; P = Pittsburgh, Pa. (industrial) 109 d.

DISCUSSION

Thus far, we have described only the phenomenological effects of pH and temperature on the stress corrosion behavior of aluminum alloys exposed to salt-(di)chromate solutions. We found that: (1) while too aggressive for a practical test, acid solutions at $\text{pH} < 1.5$ caused rapid cracking in a variety of aluminum alloys; and (2) neutral media promoted rapid failures in susceptible tempers of 2024 and 2219, but they were not very effective in tests of 7075-T6; nor were they effective in causing rapid failures of 7039. On the other hand, impressed anodic currents and neutral salt-(di)chromate solutions were successfully combined in a test which shows promise as a method of separating stress corrosion resistant and highly susceptible tempers of 2024, 2219 and 7075, and as a method for duplicating the threshold stresses obtained in 3.5% NaCl alternate immersion tests of these same products.

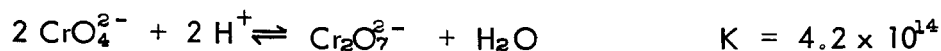
We would be remiss in our purpose, however, if we did not consider two additional requirements for a practical stress corrosion test -- simplicity and the ability to simultaneously test large numbers of specimens. Intensiostatic tests do not require elaborate fixtures or complex accessory equipment. In fact, the test method is quite simple, requiring only a battery and resistance circuit. It is necessary, however, to treat specimens individually, with spatially separated electrical circuits and cathodes. This places a physical limitation on the number of specimens which can be tested in a given area. Obviously, it would be desirable to obviate the need for impressed currents, and yet, retain the high rates of cracking required in an accelerated test. We will now briefly consider how this might be achieved.

In general, aluminum alloys act as passive electrodes in aqueous solutions because of surface oxides which have low permeability for ions, electrons or water molecules. Consequently, rupture or dissolution of these oxides has a marked effect on corrosion resistance. The oxides on aluminum can be removed in acid solutions, and at low pH the base metal is more or less in direct contact with the environment. Under these conditions, we can reasonably assume that hydrogen reduction at cathodic sites is responsible for the increased rates of corrosion or intergranular attack of aluminum alloys.

Where hydrogen ions are available only in limited quantity -- neutral or alkaline solutions-- the most probable cathodic reaction is the reduction of dissolved oxygen. In this event, dissolution of aluminum would be dependent upon the availability of oxygen at cathodic surfaces. However, corrosion rates due to oxygen reduction are generally slower than those due to hydrogen reduction because (1) the solubility of oxygen in aqueous salt solutions is low, and (2) the diffusion rate of oxygen molecules is several hundred times slower than that for hydrogen ions.

Ketchum (Ref 19) suggested that, while the initiation of intergranular attack in aluminum alloys exposed to neutral salt solutions may be dependent upon oxygen reduction, ultimately, the cathodic reaction in the crevice could change from oxygen reduction to hydrogen ion discharge (sic). Thus, during alternate immersion tests, oxygen reduction (or the action of differential aeration cells) may contribute to the initiation of stress corrosion. But this process would be slow -- accounting, in some measure, for the delay or incubation period prior to the onset of cracking. On the other hand, hydrogen reduction, being a faster cathodic reaction, probably contributes most to acceleration of intergranular attack and final rapid crack propagation. Of course, dissolved oxygen may be required to facilitate hydrogen depolarization -- but the point to be made here is that if we are to accelerate stress corrosion of aluminum alloys, we must provide an alternate reaction to oxygen reduction; specifically, hydrogen ion reduction. Moreover, if this acceleration is to be achieved in neutral solutions, we must increase the availability of hydrogen ions.

Salt-(di)chromate solutions contain chromate and dichromate ions in equilibrium according to:



There is, however, the additional equilibrium:



Thus, a half neutralized solution contains undissociated bichromic acid and chromate ions in equimolar quantities. Further, we see that a solution containing a weak partially dissociated acid has a greater buffering capacity (availability of hydrogen ions) than a strong totally dissociated acid

at a given pH. (The solution will exhibit maximum buffering capacity at about pH = 6.5.) And, finally, the neutral salt-(di)chromate solutions contain an excellent anodic inhibitor (chromate ion) which limits the general corrosion of aluminum. As a result, we must conclude that the effectiveness of these media to cause rapid stress corrosion failures in alloys 2024 and 2219 can be attributed to the availability of hydrogen ions and the inhibition of general surface corrosion.

The difference in performance of the 2000 and 7000 series alloys exposed to neutral salt-(di)chromate solutions may be related to: (1) the larger exchange current density for hydrogen reduction on the 2000 series alloys (efficient cathodes) and, therefore, intergranular attack was more rapid in these products; or (2) hydrogen depolarization was not facilitated in (non-aerated) salt-(di)chromate solutions and the small diffusion limited currents would not support rapid crack propagation in the 7000 series alloys.

Our attempt to increase the rate of intergranular attack in the 7000 series alloys by increasing the test temperature was successful only for alloy 7039. For example, the short-transverse failure times for 7039-T64 plate stressed at 35,000 psi and exposed to a neutral 6% NaCl-2% $K_2Cr_2O_7$ solution at 30C ranged from several days to more than two weeks. On the other hand, failures were observed in less than 15 minutes when these same materials were tested in a boiling solution. We suggest that these results are dramatic proof (1) of the role of hydrogen reduction in facilitating rapid intergranular attack (the probability of oxygen reduction occurring in boiling solutions is nil), and (2) that increasing temperature will increase the exchange current for anodic dissolution of 7039.

The reduction of hydrogen ions may be accelerated by dissolved oxygen. As such, the erratic stress corrosion behavior of 7075-T6 in non-aerated neutral salt-(di)chromate solutions may be attributed to a lack of oxygen (no cracking) or to oxygen depletion with time (no crack propagation). Moreover, the reduced solubility of oxygen with temperature may explain the delay in cracking of 2024-T4 and 2219-T42 and absence of cracking in 7075-T6 when tested in neutral media at temperatures at or in excess of 60C. Thus, we might find that aerated (spraying, alternate immersion, etc)

neutral salt-(di)chromate solutions are sufficiently aggressive for use in an accelerated test. There is, however, the alternate approach -- that of adding strong oxidizing agents to the test solution. And, while certain oxidizing agents may facilitate hydrogen depolarization, the possibility exists that an oxidizer will also function as an alternate cathodic reactant to hydrogen ions*.

For example, Peterson et al (Ref 20) conducted stress corrosion tests of 7079-T6 alloy in which hypochlorite ion was intentionally generated in a 3.5% NaCl solution before exposure of the specimens. While these investigations did not report the final pH of this solution (undoubtedly it was alkaline), failures of 7079 occurred in about one-third of the time required for cracking in a simple 3.5% NaCl solution at pH = 5.6 to 6.8. Hypochlorite ion is a moderately strong oxidizing agent which, interestingly, exhibits a redox potential ($E^{\circ} = 0.9 \text{ V}$) equivalent to that for oxygen reduction in neutral aerated water. As such, additions of hypochlorite ion to neutral salt-(di)chromate solutions may prove as effective, or, because of faster oxidation kinetics, more effective than dissolved oxygen in accelerating stress corrosion of aluminum alloys.

We recognize that aqueous solutions of hypochlorite ion are not particularly stable at room temperature**. As such, chlorates, nitrites, or peroxydisulfates may be more suitable for a practical test. It remains to be seen, however, if oxidation by these agents is kinetically fast enough to effect rapid stress corrosion cracking of aluminum alloys.

One final point deserves brief mention. Buffering (increasing the availability of hydrogen ions) may be an effective means for accelerating stress corrosion of aluminum alloys exposed to chloride-bearing solutions in the range of pH 4 to 7. For reasons of difficulty of control or their noxious character, buffer systems based on carbonic, sulfurous, or hydrosulfuric acids would not be acceptable. On the other hand, the buffer systems based on potassium acid citrate, acetic acid-sodium acetate, potassium acid phthalate-sodium hydroxide, and phosphoric acid-sodium hydroxide will be considered for use in our tests.

* Chromate is a moderately stronger oxidizer. Dichromate is even stronger, yet, we suspect that the direct oxidation by these ions is kinetically slow.

** Hydrogen peroxide was not considered. Not only is it unstable, but it also reduces chromates and dichromates.

STATUS

These studies under Contract NAS8-20275 will be continued for one year. Our technical program was submitted to MSFC, NASA, 15 February 1967. Therein, we considered modification of the neutralized salt-(di)chromate solutions to include additions of oxidizing agents which may (1) facilitate hydrogen depolarization, or (2) function as alternate cathodic reactants to either oxygen or hydrogen reduction. Another approach will involve the use of buffered-sodium chloride solutions in accelerated stress corrosion tests of aluminum alloys.

REFERENCES

1. H. B. Romans, "Stress Corrosion Test Environments and Test Durations", paper presented to ASTM Committee G-1 on Corrosion of Metals/VI-Stress Corrosion and Corrosion Fatigue, Task Group 2 (1966), in press. See also: F. H. Haynie and W. K. Boyd, "Stress Corrosion Cracking of Aluminum Alloys", DMIC Report 228, July 1, 1966, pp 23-24.
2. B. W. Lifka and D. O. Sprowls, "Stress Corrosion Testing of Aluminum Alloy 7079-T6 in Various Environments", paper presented at ASTM Symposium on Stress Corrosion Testing, June, 1966, in press.
3. H. B. Romans and H. L. Craig, "Factors Affecting the Rate of Stress Corrosion Cracking in an Aluminum-Zinc-Magnesium Alloy", ibid, in press.
4. G. F. Sager, R. H. Brown, and R. B. Mears, "Tests for Determining Susceptibility to Stress Corrosion Cracking", ASTM-AIME Symposium on Stress Corrosion Cracking of Metals, 1944, pp 264-267.
5. B. C. Madden, Jr., "Test Methods and Progress in the Stress Corrosion Investigation at Wright Field", ibid, pp 242-245.
6. R. B. Mears, C. J. Walton, and G. G. Eldredge, "An Alternate Immersion Test for Aluminum-Copper Alloys", Proc. ASTM, Vol. 44, 1945, pp 639-653.
7. W. H. Colner and H. T. Francis, "Influence of Exposed Area on Stress-Corrosion Cracking of 24S Aluminum Alloy", NACA Tech. Note 3292, November, 1954.
8. B. W. Lifka, W. King, M. B. Shumaker, R. A. Kelsey, and D. G. Vandenburg, "Investigation of the Stress-Corrosion Cracking of High Strength Aluminum Alloys", Summary Report NAS 8-5340, August 1, 1965, pp 22-23.
9. H. Nichols and W. Rostoker, "Analogies Between Stress Corrosion Cracking and Embrittlement by Liquid Metals", Trans, ASM, Vol. 56, September, 1963, pp 498-500.
10. E. M. Zaretskii and A. F. Kireeva, "A Rapid Method for Determining the Tendency of Duralumin Type Alloys to Corrosion Cracking", Industrial Laboratory, Vol. 29, No. 9, March, 1964, pp 1193-1196.

11. W. J. Helfrich, "Influence of Stress and Temperature on Short-Transverse Stress Corrosion Cracking of an Al-4.2% Zn-2.5% Mg Alloy", ASTM Symposium on Stress Corrosion Testing, June, 1966, in press.
12. W. Gruhl, "The Temperature Dependence for Stress Corrosion Cracking of AlZnMg3", Z. Metallkunde, Vol. 53, No. 10, October, 1962, p 670.
13. H. Nichols and W. Rostoker, "Intergranular Corrosion Penetration in an Age-Hardenable Aluminum Alloy", J. Electrochem. Soc., Vol. 112, No. 1, January, 1965, pp 108-109.
14. H. L. Logan, "Rapid Determination of Susceptibility of Aluminum Alloys to Intercrystalline Corrosion", Metal Progress, Vol. 57, February, 1950, p 211.
15. E. Herzog, "Rapid Tests for Intercrystalline Corrosion", Metal Progress, Vol. 58, September, 1950, pp 355-356.
16. S. J. Ketcham and W. Beck, "Detecting Intergranular Corrosion Susceptibility in Aluminum Alloys", Corrosion, Vol. 16, January, 1960, pp 37t-40t.
17. F. F. Booth and H. P. Godard, "An Anodic Stress Corrosion Test for Aluminum-Magnesium Alloys", First International Congress on Metallic Corrosion, London, 1961, Butterworths & Co., Ltd, London, 1962, pp 703-710.
18. M. K. Budd and F. F. Booth, "Structure-Corrosion Testing of Aluminum Alloys by Electrical Methods", presented at a Symposium on Stress Corrosion Cracking, New York, March, 1963, Second International Congress on Metallic Corrosion, NACE, 1966, pp 44-53.
19. S. J. Ketcham, "Applications of Potentiostatic and Galvanostatic Techniques to the Study of Intergranular Corrosion in High Strength Aluminum Alloys", Final Report, Project NAEC-AML(18)-R360FR101, (AD-476,857), 29 October 1965, p 13.
20. M. H. Peterson, J. A. Smith and B. F. Brown, "Effects of the Electrochemical Potential on Stress Corrosion Cracking of Aluminum Alloy 7079-T6 in Salt Water", Second International Congress on Metallic Corrosion, NACE, 1966, pp 124-127.

TABLE I
CHEMICAL COMPOSITIONS, TENSILE PROPERTIES, AND CONDUCTIVITIES
OF EXPERIMENTAL MATERIALS

Alloy	Lot No.	Chemical Composition, Weight Per Cent ¹										Zr	Ti	V
		Si	Fe	Cu	Mn	Mg	Cr	Zn						
2024	777021	0.16	0.26	4.44	0.60	1.48	0.03	0.15				0.02	---	
2024	863642	0.14	0.31	4.50	0.64	1.55	0.03	0.15				0.03	---	
2219	(a)	0.15	0.27	6.24	0.30	0.02	0.01	0.06				0.06	0.14	
7075	830882	0.14	0.29	1.68	0.13	2.71	0.19	5.59				0.04	---	
7039	742101	0.08	0.13	0.05	0.16	2.58	0.16	3.78				0.02	---	
7039	363251	0.14	0.30	0.03	0.24	2.50	0.16	4.20				0.01	---	
7039	105108	0.10	0.18	0.03	0.26	2.91	0.18	4.00				0.01	---	

Alloy-Temper	Lot No.	Thick., in.	Long Transverse ³		Short Transverse ⁴		Conductivity, ⁵ % IACS		
			TS, ksi	YS, ksi	Elong, % in 2 in.	TS, ksi		YS, ksi	Elong, % in 1 in.
2024-T4	777021	2.00	66.6	45.4	15.8	56.4	42.8	3.0	30.2-30.8
-T6	"	"	69.8	64.0	7.5	63.2	61.6	1.0	38.9-40.2
-T351	863642	2.11	66.6	48.0	13.0	55.2	43.5	3.0	29.5-30.4
-T851	"	"	70.0	63.9	6.5	64.9	62.1	1.0	38.8-39.6
2219-T42	142081 ²	2.50	50.6	22.4	24.8	48.4	21.6	12.0	28.5-29.0
-T62	"	"	61.0	41.4	11.2	58.6	43.2	5.0	33.1-33.7
-T351	142101 ²	"	54.2	37.3	20.0	49.3	30.9	12.0	28.1-28.6
-T851	"	"	69.5	55.5	9.5	65.9	54.2	5.0	32.3-33.0
-T37	142091 ²	"	57.3	43.4	15.0	52.0	37.7	8.0	28.3-28.7
-T87	"	"	71.7	60.0	9.5	66.8	57.9	4.0	31.9-32.4

7075-T651	830882	2.50	80.6	69.6	8.2	72.4	64.2	2.0	29.3-31.6
-T7351	"	"	69.7	56.8	9.5	62.3	53.8	2.0	39.4-39.7
7039-T64	742101	2.00	62.6	53.1	13.8	60.4	50.6	5.0	--
-T61	"	"	61.4	51.4	14.2	59.2	50.5	6.0	--
-T64	363251	1.00	66.1	57.5	13.0	--	--	--	--
-T6E132	105108	2.00	64.6	54.6	14.0	64.0	55.0	4.5	--

- ¹ Obtained quantometrically on plate samples.
- ² All experimental lots taken from a single 2.5-inch gage 2219-F plate.
- ³ Long-transverse tensile rounds, average of duplicate tests, 0.500-inch diameter, 4-D gage length, 1/4 T position.
- ⁴ Short-transverse tensile rounds, average of duplicate tests, 0.250-inch diameter, 4-D gage length.
- ⁵ Range of measurements taken on 3-inch centers over top and bottom surfaces of plate.

TABLE II

EFFECT OF pH AND CHLORIDE ION CONCENTRATION ON SHORT-TRANSVERSE STRESS CORROSION CRACKING OF 2024-T351, -T4 AND -T6 CONTINUOUSLY IMMERSSED IN SALT-(DI)CHROMATE SOLUTIONS AT 30C

(Etched 0.948-inch C-rings, solutions contained 20 g/l $K_2Cr_2O_7$, Cl^- added as NaCl or HCl, pH adjusted with HCl or NaOH.)

Temper (Lot No.)	Stress, ksi	pH	F/N ¹	Failure Time
<u>Part A, 1.0 M Cl^-</u>				
-T351 (863642)	35	1.0	3/3	23 min
"	"	2.0	"	50-60 min
"	"	3.0	"	11, 48, 48 hr
"	"	3.7	"	8-17 hr
"	"	5.0	"	14-150 min
"	"	6.0	"	10-55 min
"	"	7.0	"	10-20 min
-T4 (777021)	35	1.0	3/3	10-15 min
"	"	2.0	"	50-60 min
"	"	3.0	"	21, 48, 52 hr
"	"	3.7	"	8-17 hr; 9, 18, 20 hr (repeat)
"	"	5.0	"	80-150 min
"	"	6.0	"	8-55 min
"	"	6.9	"	9 min
-T6 (777021)	35	6.9	0/3	> 14 d
"	45	"	1/3	2, > 14 d
"	55	"	2/3	1, 2, > 14 d
<u>Part B, 2.0 M Cl^-</u>				
-T4 (777021)	35	7.0	3/3	20-40 min
-T6 (777021)	"	7.0	3/3	4, 7, 7d
"	45	"	"	1, 4, 5 d
"	55	"	"	2-31 hr
<u>Part C, 5.0 M Cl^-</u>				
-T4 (777021)	35	7.0	3/3	50-60 min
-T6 (777021)	35	7.0	3/3	3-19 hr
"	45	"	"	3-19 hr
"	55	"	"	3-19 hr

¹ Number of failures/number of specimens tested.

TABLE III
EFFECT OF pH ON SHORT-TRANSVERSE STRESS CORROSION CRACKING OF
2219-T42 CONTINUOUSLY IMMERSSED IN SALT-(DI)CHROMATE
SOLUTIONS AT 30C

(Etched 0.948-inch C-rings, stressed 20 ksi, solutions contained 20 g/l $K_2Cr_2O_7$,
1.0 M Cl^- , pH adjusted with HCl or NaOH.)

pH	F/N ¹	Failure Time
<u>Lot 142081</u>		
1.0	3/3	60-120 min
2.0	2/3	4, 4, > 7d
3.0	1/3	7, > 7d
3.7	3/3	35, 46 hr; 7 d
5.0	3/3	250-320 min
7.0	3/3	45-100 min

¹ Number of failures/number of specimens tested.

TABLE IV
EFFECT OF pH ON SHORT-TRANSVERSE STRESS CORROSION CRACKING OF
7075-T651 CONTINUOUSLY IMMERSSED IN SALT-(DI)CHROMATE
SOLUTIONS AT 30C

(Etched 0.948-inch C-rings, stressed 45 ksi, solutions contained 20 g/l $K_2Cr_2O_7$,
1.0 M Cl^- , pH adjusted with HCl or NaOH.)

<u>pH</u>	<u>F/N¹</u>	<u>Failure Time</u>
<u>Lot 380882</u>		
1.0	3/3	10-30 min
3.0	3/3	40-60 min
3.7	3/3	25-90 min
4.5	0/3	> 5 d
5.0	0/3	> 5 d
6.0	0/3	> 5 d
6.5	0/3	> 5 d
7.0	3/3	60 min (to > 5 d) ²

¹ Number of failures/number of specimens tested.

² Repeat tests revealed wide scatter in these data, see text.

TABLE V
EFFECT OF pH ON SHORT-TRANSVERSE STRESS CORROSION CRACKING OF
7039-T64 CONTINUOUSLY IMMERSSED IN SALT-(DI)CHROMATE
SOLUTIONS AT 30C

(Etched 0.948-inch C-rings, stressed 35 ksi, solutions contained 20 g/l
K₂Cr₂O₇, 1.0 M Cl⁻, pH adjusted with HCl or NaOH.)

<u>pH</u>	<u>F/N¹</u>	<u>Failure Time</u>
<u>Lot 363251</u>		
1.0	3/3	8, 8, 24 hr
3.7	3/3	17, 19, 19 hr
6.0	3/3	32, 43, 43 hr
6.9	3/3	2, 4, 5 d
<u>Lot 742101</u>		
1.0	3/3	20-24 hr
3.7	3/3	43, 46, 46 hr
6.0	2/3	6, 7, > 14 d
6.9	0/3	> 14 d

¹ Number of failures/number of specimens tested.

TABLE VI
INFLUENCE OF TEMPERATURE ON SHORT-TRANSVERSE STRESS CORROSION
CRACKING OF 7039-T64, -T61 AND -T6E132 SUBJECT
TO CONTINUOUS IMMERSION IN 1 MOLAR NaCl SOLUTION

(Etched 0.948-inch C-rings, stressed at 35 ksi, solution pH = 6.0-6.5.)

Temper (Lot No.)	Test Temp, C	F/N ¹	Time to Failure
-T64 (363251)	30	3/3	20-28 hr
"	60	3/3	60-90 min
"	100	3/3	5 min
-T64 (742101)	30	3/3	43, 51 hr; 7 d
"	60	3/3	110-128 min
"	100	3/3	8 min
-T6E132(105108)	30	3/3	67 hr; 7, 14 d
"	60	3/3	80-144 min
"	100	3/3	5 min
-T61 (742101)	30	3/3	1, 4, 7 d
"	60	3/3	76-117 min
"	100	3/3	8 min

¹ Number of failures/number of specimens tested.

TABLE VII
EFFECT OF TEMPERATURE ON SHORT-TRANSVERSE STRESS CORROSION OF
2024-T4 AND 2219-T42 SUBJECT TO CONTINUOUS IMMERSION
IN SALT-(DI)CHROMATE SOLUTIONS AT pH=1, 3.7 AND 7

(Etched 0.948-inch C-rings, solutions contained 20 g/l $K_2Cr_2O_7$, 1.0 M Cl^- ,
pH adjusted with HCl or NaOH.)

pH	Test Temp, C	F/N ¹	Time to Failure
<u>2024-T4, Stressed 35 ksi</u>			
1.0	0	3/3	30-60 min
	30	3/3	15 min
	60	3/3	20-45 min
	100	3/3	10 min
3.7	0	1/3	7, > 16 d
	30	3/3	9-20 hr
	60	3/3	80-91 min
	100	3/3	9 min
6.9	0	0/3	> 7 d
	30	3/3	9 min
	60	3/3	7 min
	100	3/3	20, 30 min; 4 hr
<u>2219-T42, Stressed 20 ksi</u>			
7.0	30	3/3	45-100 min
	60	3/3	18 min
	100	0/3	> 3 d

¹ Number of failures/number of specimens tested.

TABLE VIII
INFLUENCE OF TEMPERATURE ON SHORT-TRANSVERSE STRESS CORROSION OF
7075-T651 SUBJECT TO CONTINUOUS IMMERSION IN SALT-(DI)CHROMATE
SOLUTIONS AT pH=1, 3.7 AND 7

(Etched 0.948-inch C-rings, stressed 45 ksi, lot 830882, solutions contained 20 g/l $K_2Cr_2O_7$, 1.0 M Cl^- , pH adjusted with HCl or NaOH.)

pH	Test Temp, C	F/N ¹	Time to Failure
1.0	0	3/3	2-4 hr
	30	3/3	15-30 min
	60	0/3	> 2 hr
	100	0/3	> 2 hr
3.7	0	0/3	> 15 d
	15	3/3	2 hr
	30	3/3	25-90 min
	60	0/3	> 66 hr
	100	2/3	6, 18 hr, > 1 d
	100	0/3	> 1 d
7.0	60	0/3	> 1 d
	100	0/3	> 1 d

¹ Number of failures/number of specimens tested.

TABLE IX
EFFECT OF TEMPERATURE ON SHORT-TRANSVERSE STRESS CORROSION
OF 7039-T64 CONTINUOUSLY IMMERSSED IN SALT-(DI)CHROMATE
SOLUTIONS AT pH = 1, 3.7 AND 7

(Etched 0.948-inch C-rings, stressed 35 ksi, solutions contained 20 g/l
 $K_2Cr_2O_7$, 1.0 M Cl^- , pH adjusted with HCl or NaOH.)

pH	Test Temp, C	F/N ¹	Time to Failure
<hr/>			
Lot 363251			
1.0	0	3/3	4, 11, 11 d
	30	3/3	19, 19, 24 hr
	60	3/3	26-34 min
	100	3/3	3 min
3.7	30	3/3	17-19 hr
	60	3/3	42-55 min
	100	3/3	3 min
7.0	30	3/3	2, 4, 5 d
	60	3/3	4, 20, 20 hr
	100	3/3	10 min
<hr/>			
Lot 742101			
1.0	0	2/3	11, 11, >20 d
	30	3/3	1 d
	60	3/3	70-140 min
	100	3/3	7 min
3.7	30	3/3	43-46 hr
	60	3/3	135-175 min
	100	3/3	11 min
7.0	30	0/3	> 14 d
	60	3/3	12, 12, 45 hr
	100	3/3	14 min

¹ Number of failures/ number of specimens tested.

TABLE X
SHORT-TRANSVERSE STRESS CORROSION OF COPPER-ALUMINUM ALLOY
GALVANIC COUPLES IN A NEUTRAL 5% NaCl-2% K₂Cr₂O₇ SOLUTION AT 30C
(Etched 0.948-inch diameter C-rings coupled to high-purity copper foil. All but the upper tension and compression surfaces of the C-ring and 2 cm² of copper were coated with 5% polyethylene in paraffin wax.)

Alloy-Temper (Lot No.)	Applied Stress, ksi	F/N ¹	Time to Failure ²
2024-T4 (777021)	35	3/3	1 hr
2024-T6 (777021)	55	3/3	6, 17, 17 hr
"	45	3/3	17, 41 hr; 5 d
"	35	1/3	5 d
2024-T351 (863642)	35	3/3	1 hr
2024-T851 (863642)	55	3/3	23, 41, 41 hr
"	45	1/3	3 d
"	35	0/3	
2219-T42 (142081)	20	3/3	1 hr
2219-T62 (142081)	35	0/3	
"	25	0/3	
2219-T351 (142101)	30	3/3	1 hr
2219-T851 (142101)	50	0/3	
"	40	0/3	
2219-T37 (142091)	35	3/3	1 hr
2219-T87 (142091)	55	1/3	3 d
"	45	1/3	1 d
7075-T651 (830882)	45	3/3	1 hr
7075-T7351 (830882)	50	0/3	
"	45	0/3	
7039-T64 (363251)	35	3/3	1 d
7039-T64 (742101)	35	3/3	2 d
7039-T61 (742101)	35	3/3	1, 2, 2d
7039-T6E132 (105108)	35	3/3	2, 2, 2 d

¹ F/N = Number of failure/number of specimens tested.

² Test terminated after 7 days.

TABLE XII (CONT'D)

Alloy-Temper (Lot No.)	Applied Stress, ksi	F/N ¹	Time to Failure ²
2219-T351 (142101)	30	5/5	17(3), 21(2) hr
"	20	5/5	21, 23, 44 hr; 3(2) d
"	15	5/5	40 hr; 12(2), 15, 20 d
"	10	5/5	15, 23, 30(3) d
"	5	3/5	15, 30(2) d
2219-T851 (142101)	50	1/5	30 d
"	45	0/5	> 30 d
"	40	0/5	"
"	35	0/5	"
2219-T37 (142091)	35	5/5	17(3), 21(2) hr
"	30	5/5	17, 21(2), 44 hr; 6 d
"	20	5/5	21(3) hr; 15, 20 d
"	15	5/5	23 hr; 3, 16(2) d
"	10	5/5	15, 30(4) d
"	5	5/5	15, 30(4) d
2219-T87 (142091)	55	3/5	30 d
"	50	2/5	"
"	45	2/5	"
"	40	0/5	> 30 d
7075-T651 (830882)	50	9/9	1 hr
"	40	9/9	1 hr
"	30	9/9	1-2 hr
"	20	9/9	1(5), 2, 10(3) hr
"	15	9/9	5(3), 6, 10(4) hr; 4 d
"	10	9/9	30 hr; 14, 30, 32, 40(2), 60, 90(2) d
"	5	0/9	> 90 d
7075-T7351 (830882)	50	3/9	90 d
"	45	0/9	> 90 d
"	40	0/9	"
"	35	0/9	"
"	30	0/9	"
7039-T64 (363251)	40	9/9	22, 44 hr; 5(2), 6, 7(3), 9 d
"	30	9/9	8, 9(3), 11(2), 14(2), 21 d
"	20	9/9	13, 18, 21(2), 24, 45, 79, 85, 133 d

¹ F/N = Number of failures/number of specimens tested.

² Time to failure (number of specimens failed).

TABLE XII (CONT'D)

Alloy-Temper (Lot No.)	Applied Stress, ksi	F/N ¹	Time to Failure ²
7039-T64 (363251)	15	5/9	18,50,91,94,121 d
"	10	0/9	> 184 d *
"	5	0/9	"
7039-T64 (742101)	40	9/9	9,23,33(4),35,40,74 d
"	30	8/9	48,50,54,61,82,84,103,287 d
"	20	4/9	48,54,128,188d
"	15	1/9	68 d
"	10	1/9	194 d
"	5	0/9	> 287 d*
7039-T61 (742101)	40	9/9	22,29,41,42,48,63,82(2),84 d
"	30	4/9	48,97(2), 121d
"	20	0/9	> 287 d*
"	15	0/9	"
"	10	0/9	"
"	5	0/9	"
7039-T6E132 (105108)	40	9/9	19,22,27,33,36,42,48,57,74 d
"	30	9/9	36,48,82,89,121(2),138,153,250 d
"	20	1/9	275 d
"	15	0/9	> 287 d *
"	10	0/9	"
"	5	0/9	"

¹ F/N = Number of failures/number of specimens tested.

² Time to failure (number of specimens failed).

* Through 1 March 1967, tests continuing.

TABLE XI
SHORT-TRANSVERSE STRESS CORROSION OF 2024, 2219 AND 7075 TESTED
INTENSIOSTATICALLY IN NEUTRAL 3% NaCl-2% K₂Cr₂O₇ SOLUTION AT 30C

(Etched 0.948-inch diameter C-rings. All surfaces except about 2 cm² of the tension surface of each C-ring were coated with 5% polyethylene in paraffin wax. Impressed anodic current = 50 microamperes/cm².)

<u>Alloy-Temper (Lot No.)</u>	<u>Applied Stress, ksi</u>	<u>F/N¹</u>	<u>Time to Failure²</u>
2024-T4 (777021)	30	2/2	1 hr
-T6 (777021)	50	2/2	1 d
"	40	2/2	1, 4 d
"	30	0/2	
2024-T351 (863642)	30	2/2	1 hr
2024-T851 (863642)	50	2/2	1, 3 d
"	40	2/2	2, 3 d
"	30	0/2	
2219-T42 (142081)	20	1/1	1 hr
2219-T62 (142081)	35	0/2	
2219-T351(142101)	30	1/1	1 hr
2219-T851 (142101)	50	0/2	
"	40	0/2	
2219-T37 (142091)	35	1/1	1 hr
2219-T87 (142091)	55	1/2	1 d
"	45	0/2	
7075-T651(830882)	40	2/2	1 hr
7075-T7351 (830882)	50	1/2	3 d
"	45	0/2	

¹ F/N = Number of failures/number of specimens tested.

² Test terminated after seven days.

TABLE XII

SHORT-TRANSVERSE STRESS CORROSION CRACKING OF 2024, 2219, 7075 AND
7039 IN 3.5% NaCl SOLUTION BY ALTERNATE IMMERSION AT 75F \pm 2F

(0.948-inch short-transverse, C-rings, etched 30 sec in 5% NaOH at 170F, desmutted
in cold conc HNO₃.)

Alloy-Temper (Lot No.)	Applied Stress, ksi	F/N ¹	Time to Failure ²
2024-T351 (863642)	40	9/9	5-21 hr
"	30	9/9	5-21 hr
"	20	9/9	5-21 (7) hr; 1, 1d
"	15	9/9	25, 46(2), 74(2), 105(3) hr; 30 d
"	10	7/9	4(2), 5, 6, 90(3) d
"	5	0/9	> 90 d
2024-T851 (863642)	50	7/9	100(3) hr; 6, 90 (3) d
"	45	3/9	20, 113 hr; 60 d
"	40	3/9	113(2) hr; 60 d
"	35	0/9	> 90 d
"	30	0/9	> 90 d
2024-T4 (777021)	40	9/9	5-21 hr
"	30	9/9	5-21 hr
"	20	9/9	5-21(5), 27, 34(2), 45 hr
"	15	6/9	45, 51, 73 hr; 90(3) d
"	10	3/9	90 d
"	5	0/9	> 90 d
2024-T6 (777021)	50	9/9	48, 79(4), 92(2) hr; 60(2) d
"	45	7/9	26, 79, 92 (3) hr; 60(2) d
"	40	2/9	7(2) d
"	35	0/9	> 90 d
"	30	0/9	> 90 d
2219-T42 (142081)	20	5/5	40, 44 hr; 6(3) d
"	15	5/5	6, 12(3); 14 d
"	10	5/5	15 d
"	5	5/5	20, 23, 30(3) d
2219-T62 (142081)	35	0/5	> 30 d
"	30	0/5	"
"	25	0/5	"

¹ F/N = Number of failures/number of specimens tested.

² Time to failure (number of specimens failed).

TABLE XIII
SHORT-TRANSVERSE STRESS CORROSION OF 2024, 2219, 7075 AND 7039 ALLOYS
IN THE MARINE ATMOSPHERE OF DAYTONA BEACH, FLORIDA
(0.948-inch diameter C-rings, etched 30 sec in 5% NaOH at 170F, desmutted in cold
conc HNO₃ .)

Alloy-Temper (Lot No.)	Applied Stress, ksi	F/N ¹	Time to Failure, days ²
2024-T4 (777021)	40	5/5	2 (5)
"	30	5/5	2, 5(2), 13(2)
"	20	5/5	2(2), 5, 8, 13
"	15	3/5	2, 14(2)
"	10	1/5	14
"	5	0/5	
2024-T351 (863642)	40	5/5	2 (5)
"	30	5/5	2(3), 5(2)
"	20	5/5	5(3), 12(2)
"	15	4/5	14(2), 16(2)
"	10	2/5	14, 33
"	5	0/5	
2219-T42 (142081)	20	5/5	2(2), 8(2), 15
"	15	3/5	2(2), 21
"	10	2/5	8, 16
"	5	0/5	
2219-T351 (142101)	30	5/5	2(2), 8, 13(2)
"	20	1/5	28
"	15	0/5	
"	10	0/5	
"	5	0/5	
2219-T37 (142091)	35	4/5	2(3), 13
"	30	4/5	2(4)
"	20	1/5	16
"	15	0/5	
"	10	0/5	
"	5	0/5	
7075-T651 (830882)	50	5/5	2(5)
"	40	5/5	2(5)
"	30	5/5	2(5)
"	20	5/5	2(5)
"	15	5/5	2(5)
"	10	5/5	7, 13, 14(3)
"	5	0/5	

¹ F/N = Number of failures/number of specimens tested.

² Time to failure (number of specimens failed). Tests initiated October 5, 1966.
Specimens in test 117 days as of 1 March 1967.

TABLE XIII (CONT'D)

Alloy-Temper (Lot No.)	Applied Stress, ksi	F/N ¹	Time to Failure, days ²
7039-T64 (363251)	40	5/5	1(4), 2
"	30	5/5	1(4), 2
"	20	5/5	2,5,7,13,33
"	15	5/5	1,21,42,44,103
"	10	4/5	2,35,40,124
"	5	0/5	
7039-T64 (742101)	40	5/5	2(5)
"	30	5/5	2(3), 7(2)
"	20	5/5	2, 13, 56(2), 58
"	15	3/5	90(2), 96
"	10	0/5	
"	5	0/5	
7039-T61 (742101)	40	5/5	2(4), 7
"	30	5/5	2,7(2),8,9
"	20	3/5	2(2),69
"	15	0/5	
"	10	0/5	
"	5	0/5	
7039-T6E132 (105108)	40	5/5	2,7,8(3)
"	30	5/5	8,12,19,26,44
"	20	0/5	
"	15	0/5	
"	10	0/5	
"	5	0/5	

¹ F/N = Number of failures/number of specimens tested.

² Time to failure (number of specimens failed). Tests initiated October 5, 1966.
Specimens in test 117 days as of 1 March 1967.

TABLE XIV
SHORT-TRANSVERSE STRESS CORROSION OF 7075 AND 7039 IN THE
INDUSTRIAL ATMOSPHERE OF PITTSBURGH, PA.

(0.948-inch diameter C-rings, etched 30 sec in 5% NaOH at 170F,
desmutted in cold conc HNO₃ .)

Alloy-Temper (Lot No.)	Applied Stress, ksi	F/N ¹	Time to Failure, days ²
7075-T651 (830882)	50	5/5	92(3), 119(2)
"	40	5/5	92(2), 119(3)
"	30	3/5	92(2), 119
"	20	2/5	119(2)
"	15	0/5	
"	10	0/5	
"	5	0/5	
7039-T64 (363251)	40	5/5	14(3), 21(2)
"	30	5/5	14, 21, 43(3)
"	20	5/5	21, 43, 86(2), 92
"	15	5/5	43, 86(2), 92(2)
"	10	4/5	21, 92, 119(2)
"	5	0/5	
7039-T64 (742101)	40	5/5	14(2), 21(2), 43
"	30	5/5	86(4), 92
"	20	5/5	86, 92(4)
"	15	4/5	119(4)
"	10	0/5	
"	5	0/5	
7039-T61 (742101)	40	5/5	14, 86(3), 92
"	30	5/5	21, 86(3), 92
"	20	3/5	86, 92, 119
"	15	0/5	
"	10	0/5	
"	5	0/5	
7039-T6E132 (105108)	40	5/5	43(5)
"	30	5/5	86(4), 92
"	20	5/5	86(5)
"	15	3/5	86(2), 119
"	10	0/5	
"	5	0/5	

¹ N/F = Number of failures/number of specimens tested.

² Time to failure (number of specimens failed). Tests initiated 13 October 1966.
Specimens in test 109 days as of 1 March, 1967.